

# METALLURGIA

THE BRITISH JOURNAL OF METALS

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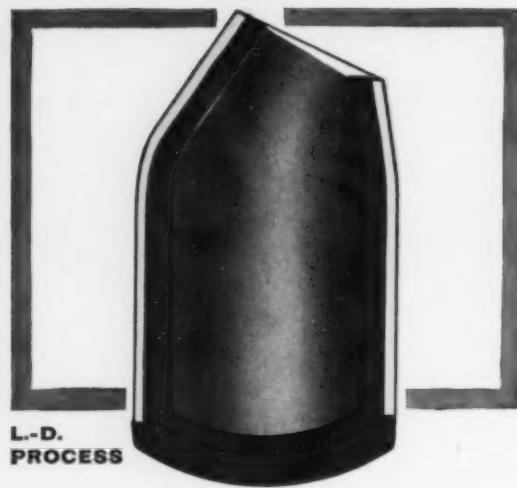
Monthly: Two Shillings and Sixpence

## HALE *Malleable Iron* CASTINGS

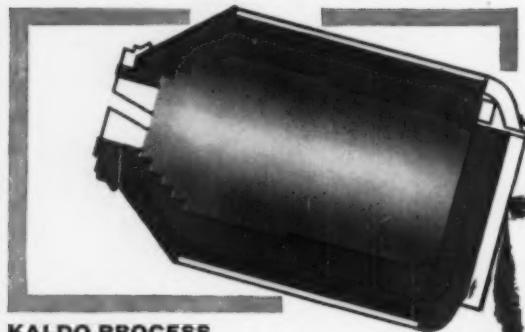


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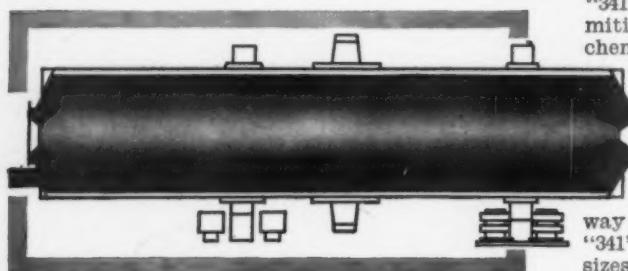
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for  
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STEELMAKING

"341" DOLOMITE bricks are manufactured from a dolomitic limestone which in the natural state approaches in chemical composition the mineral dolomite  $\text{CaCO}_3$ ,  $\text{MgCO}_3$ . The raw dolomite is dead-burned at high temperatures and it is then formed into suitable shapes by a new and unique brick-making process. Tunnel kiln firing at high temperatures produces a dense, volume stable and truly basic refractory of exceptional quality.

"341" DOLOMITE bricks are resistant to hydration and they can be transported and stocked in the same way as other basic bricks.

"341" DOLOMITE bricks are manufactured in various sizes designed to suit many applications.

#### Typical Chemical Analysis

| $\text{SiO}_2$ | $\text{TiO}_2$ | $\text{Fe}_2\text{O}_3$ | $\text{MnO}$ | $\text{Al}_2\text{O}_3$ | $\text{CaO}$ | $\text{MgO}$ |
|----------------|----------------|-------------------------|--------------|-------------------------|--------------|--------------|
| 2/3%           | 0.2%           | 1/2%                    | 0.1%         | 1/2%                    | 48/50%       | 36/40%       |

#### Typical Physical Properties

|                               |                        |   |                              |
|-------------------------------|------------------------|---|------------------------------|
| TRUE POROSITY %               | 18/20                  | SPALLING INDEX<br>(small prism test)            | +30                          |
| BULK DENSITY grams per c.c.   | 2.70/2.75              | PERMANENT LINEAR CHANGE<br>2 hrs. at 1,500° C.  | Nil to 0.2%                  |
| SPECIFIC GRAVITY              | 3.35                   | WEIGHT PER CUBIC FOOT                           | 175 lbs.                     |
| SPECIFIC HEAT                 | 0.25                   | WEIGHT PER CUBIC METRE                          | 2,700 kilos                  |
| PERMEABILITY c.g.s units      | 0.05                   | WEIGHT PER 1,000 BRICKS<br>9" x 4½" x 3" approx | 11,700 lbs.<br>" 5,300 kilos |
| COLD CRUSHING STRENGTH p.s.i. | +8,500<br>Kilos sq.cm. |   |                              |
|                               | + 600                  |   |                              |

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exclusively through  
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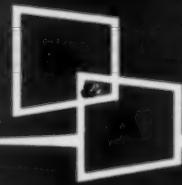
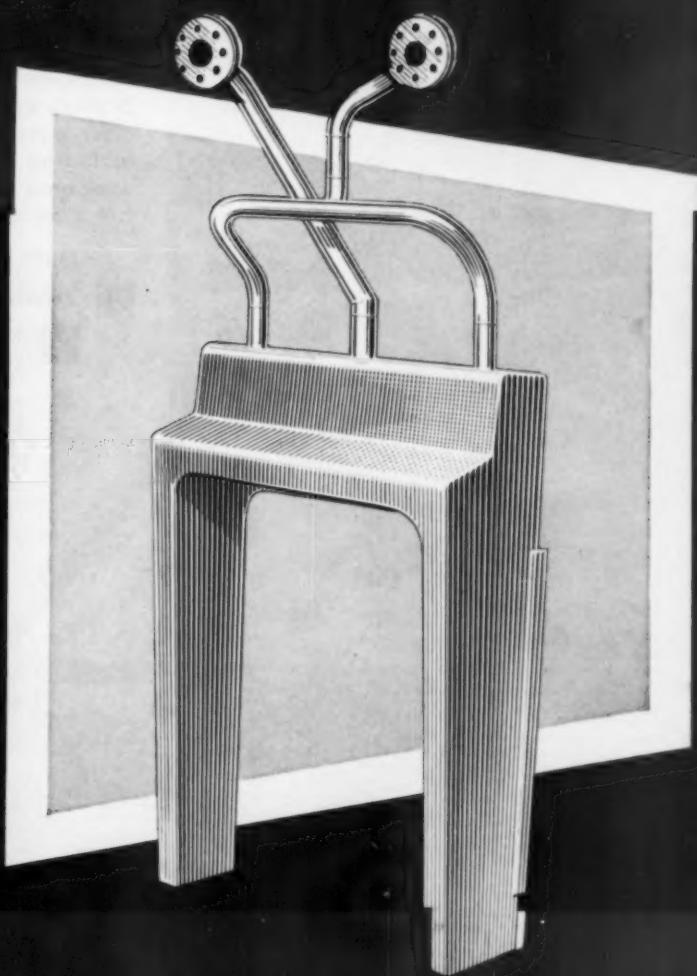
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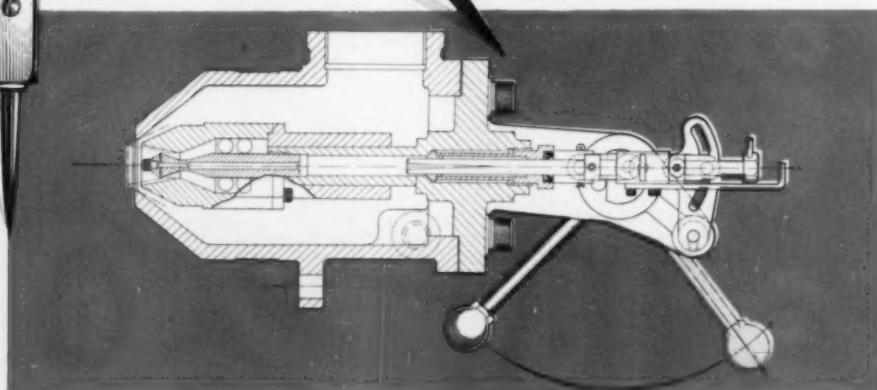
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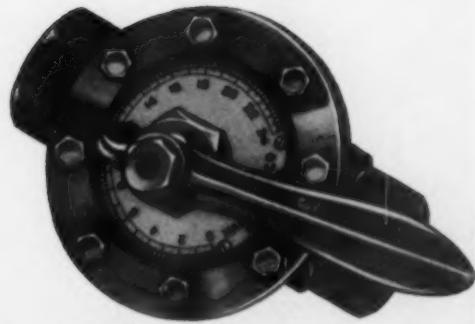
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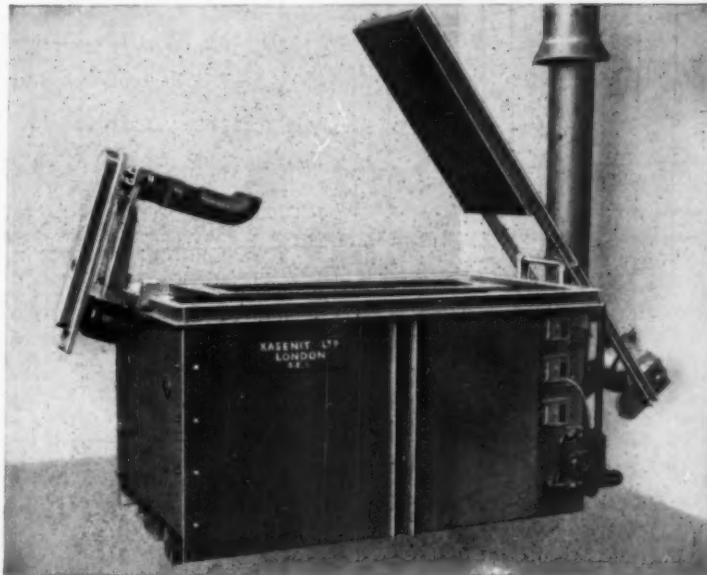
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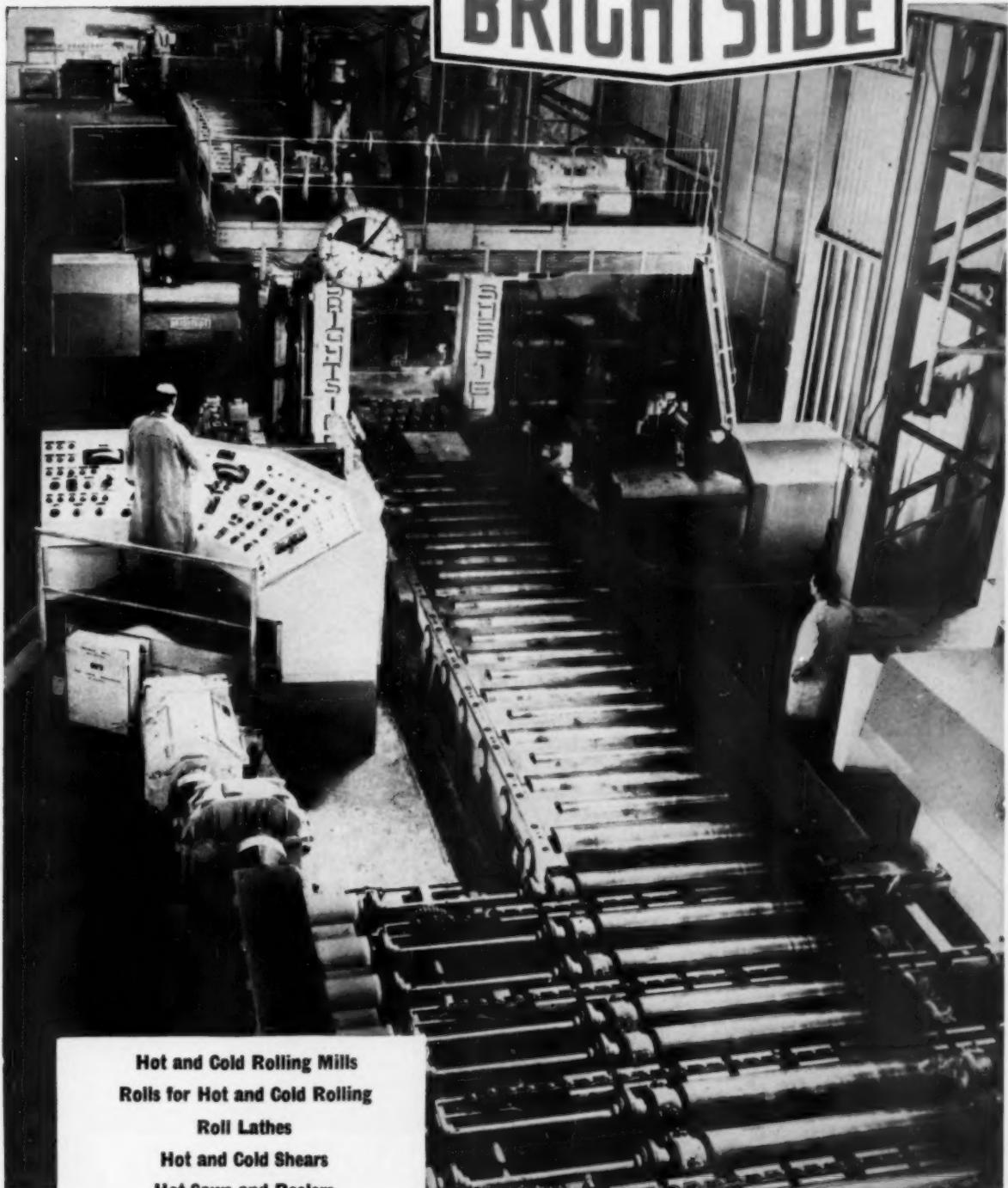
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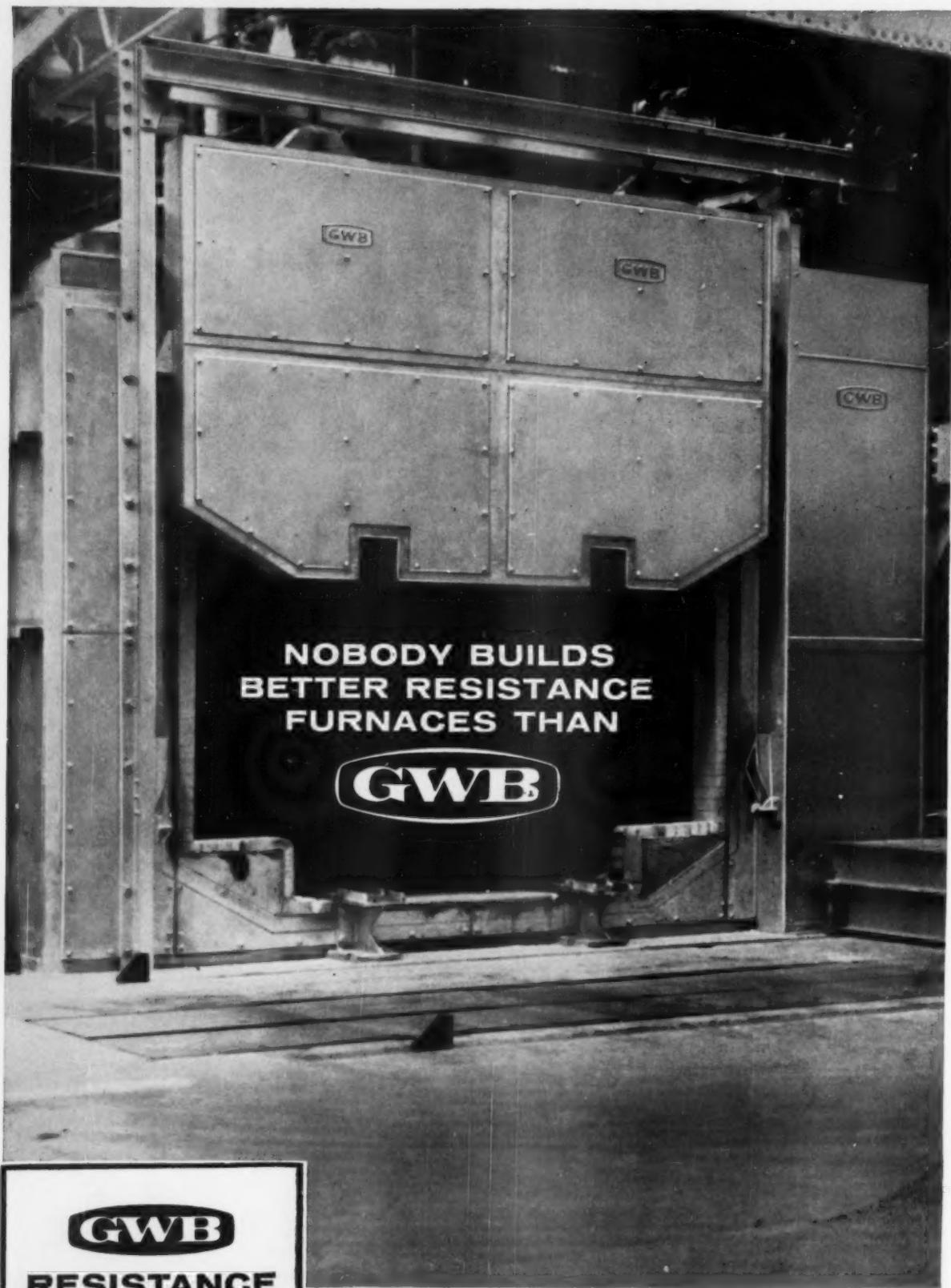
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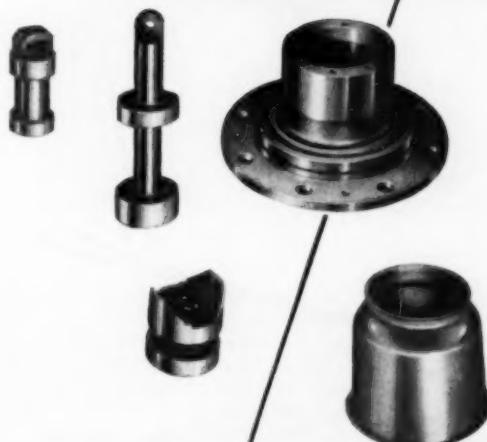
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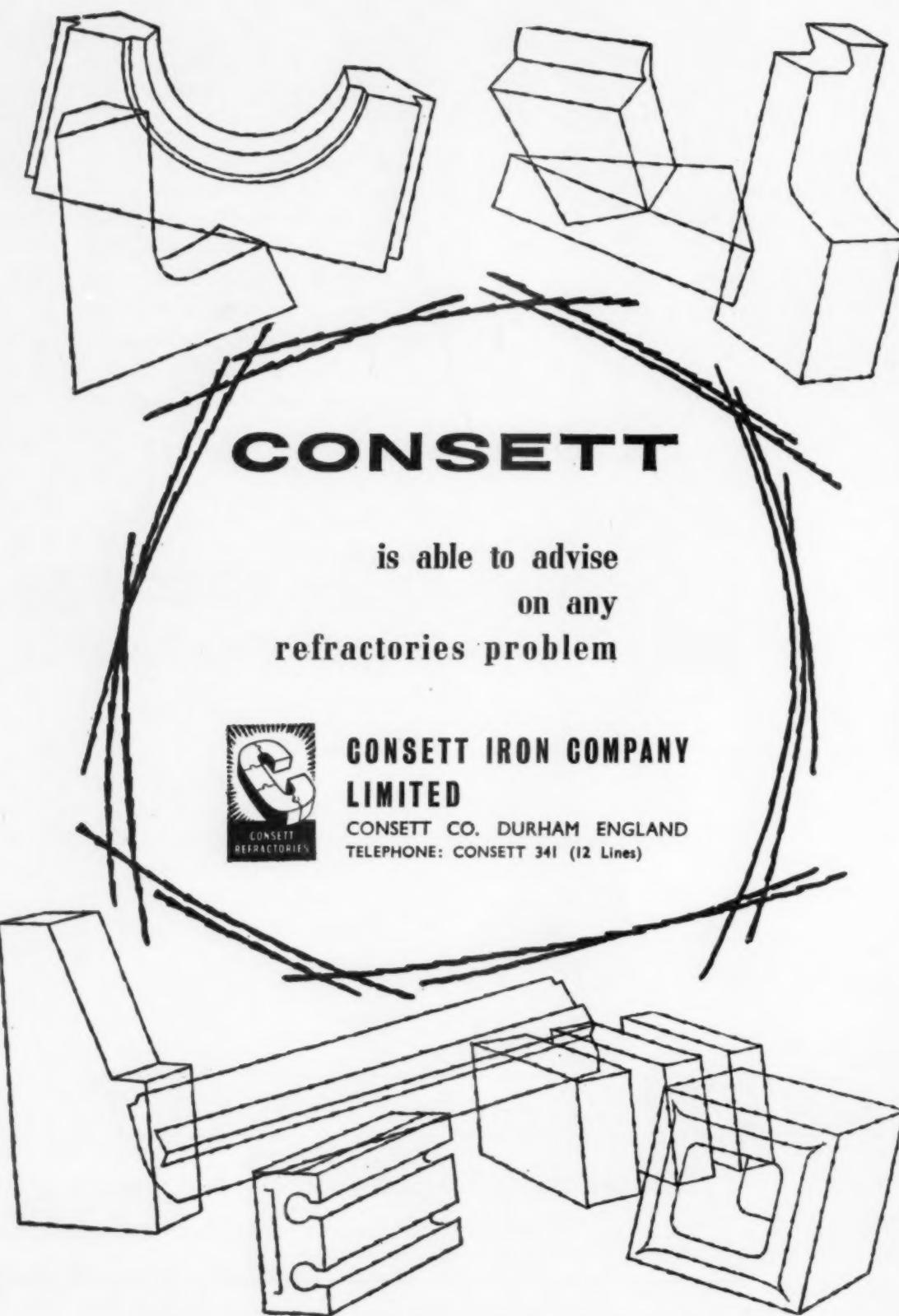
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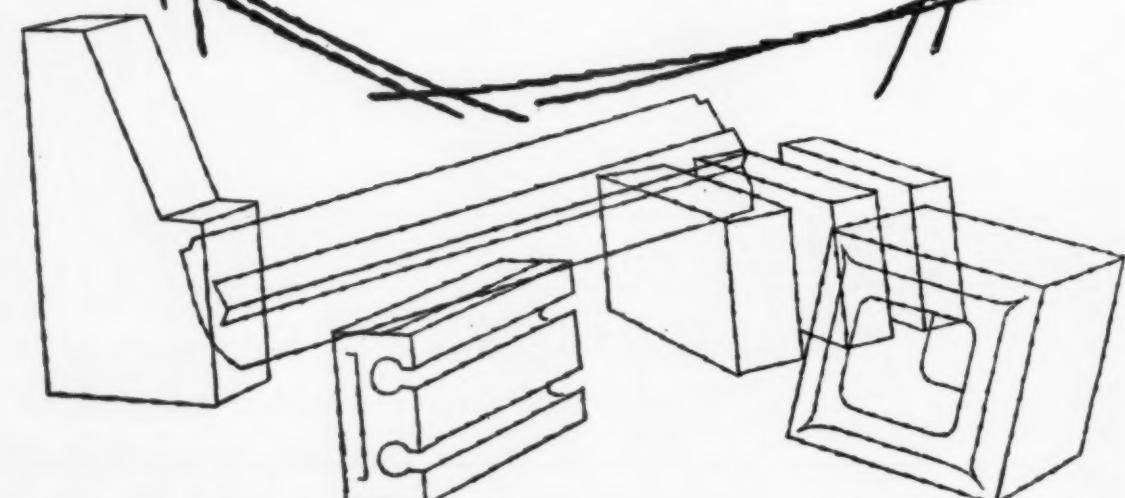
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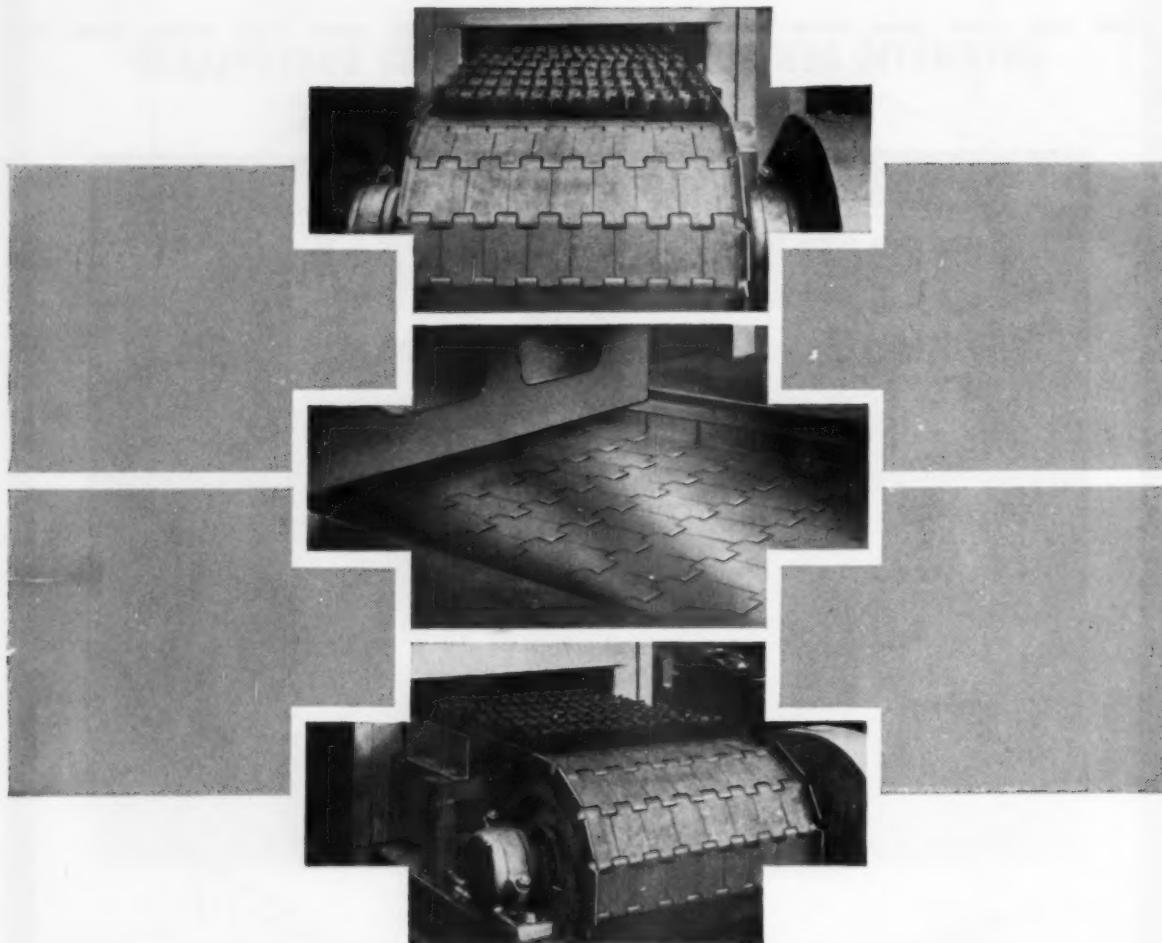


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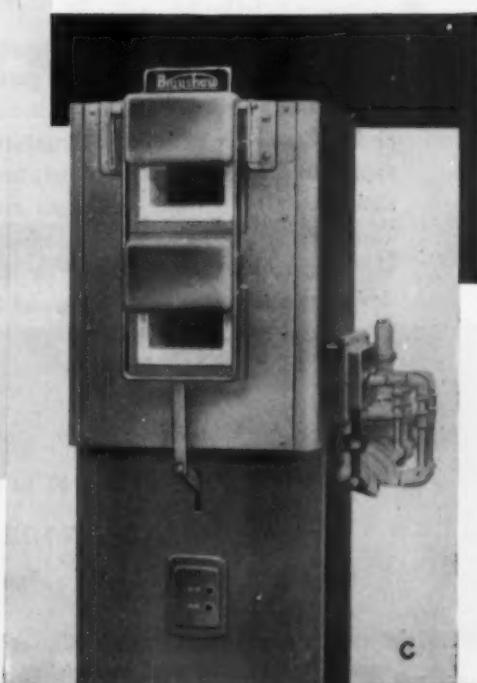
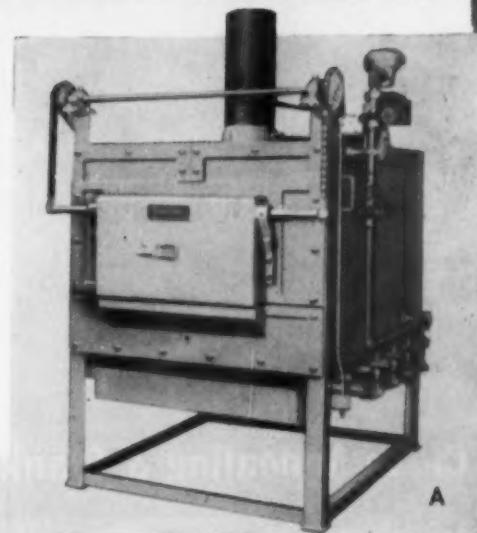
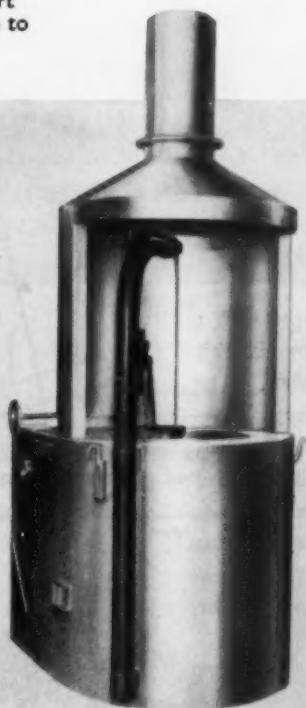
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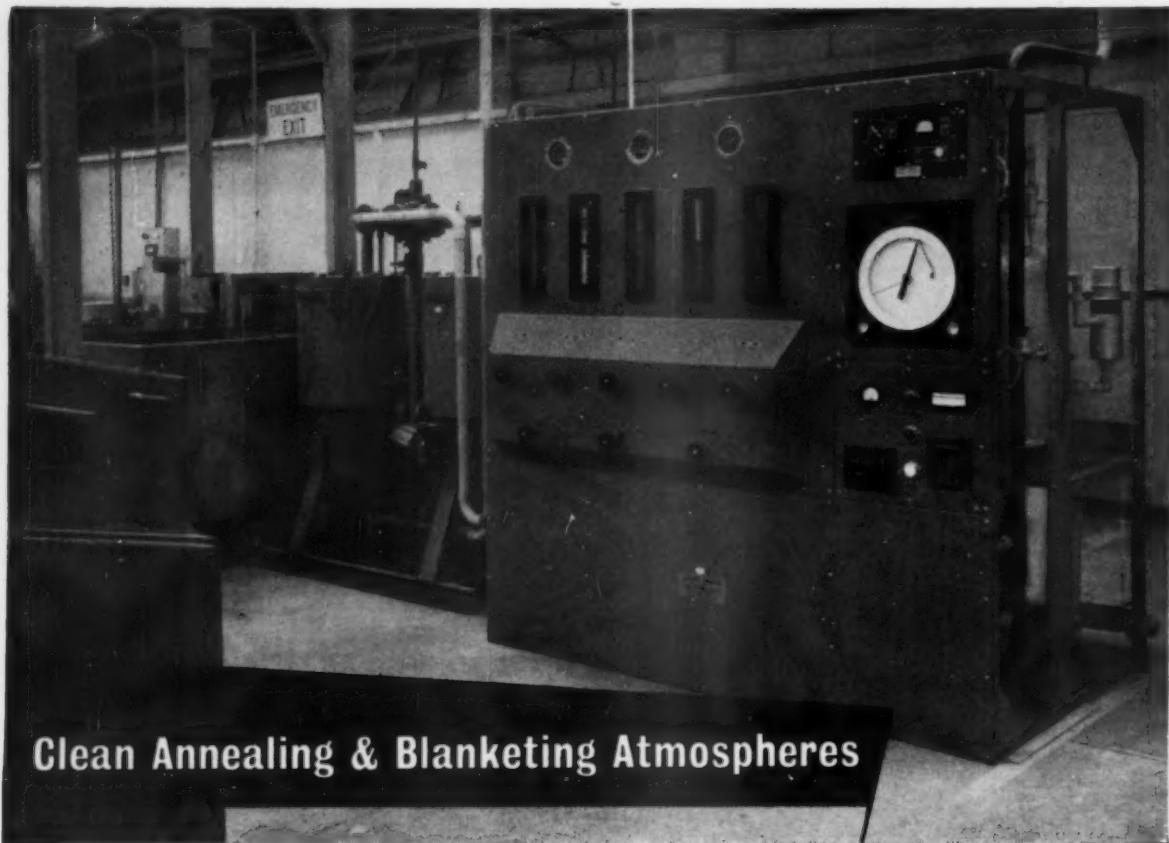
### What the illustrations show:

- A.—'Hynor' Oven Furnace.
- B.—Liquid Bath Furnace.
- C.—Twin Chambered High Speed Steel Furnace.

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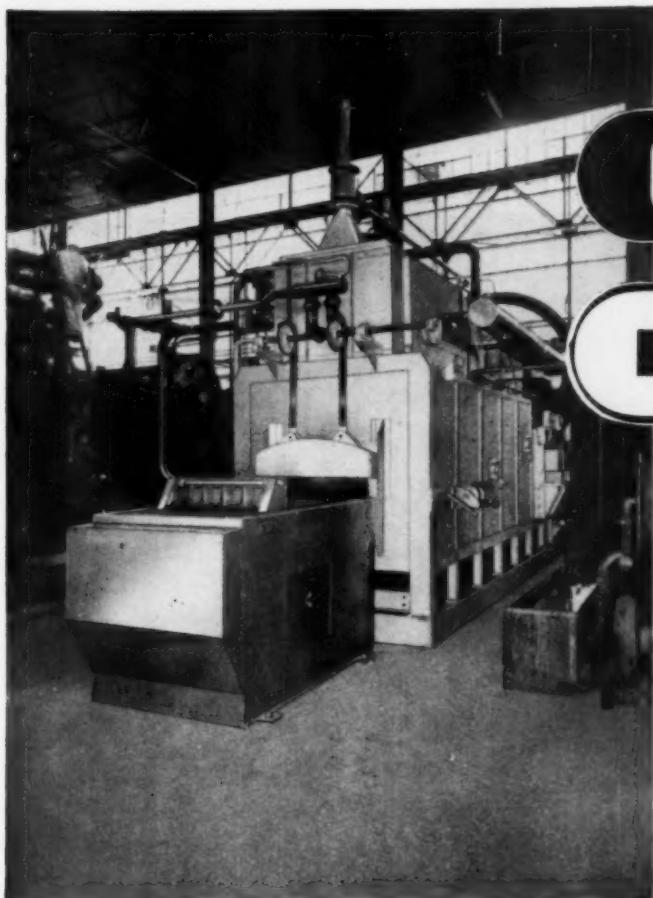
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*A view of a 1500 c.f.h. Nitroneal Generator at the Uddington Works of Ranco Limited.*

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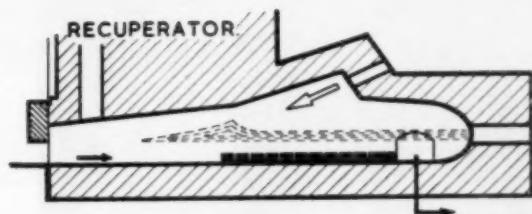
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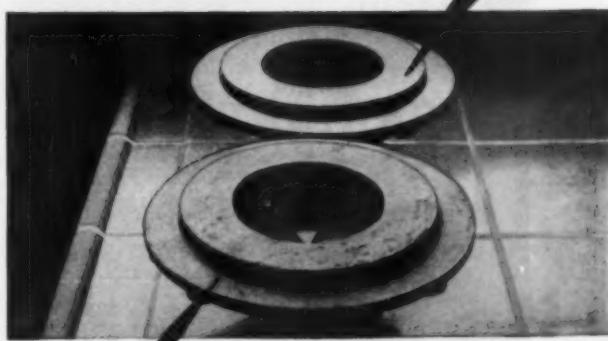


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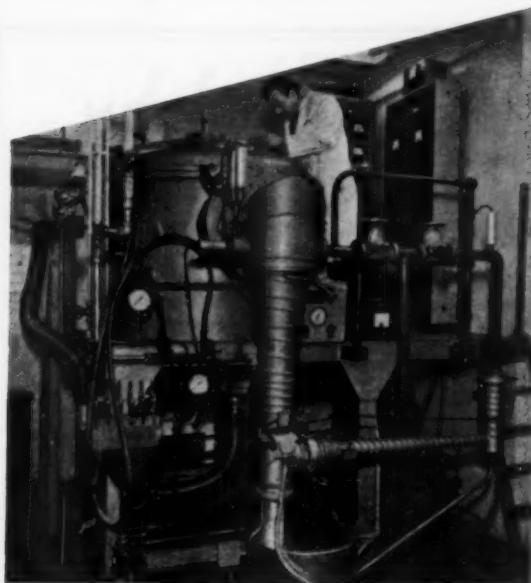
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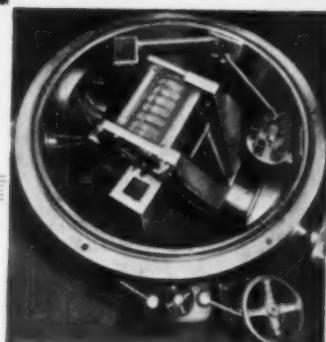


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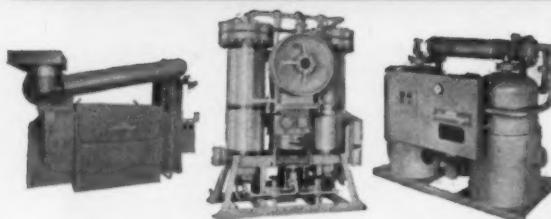
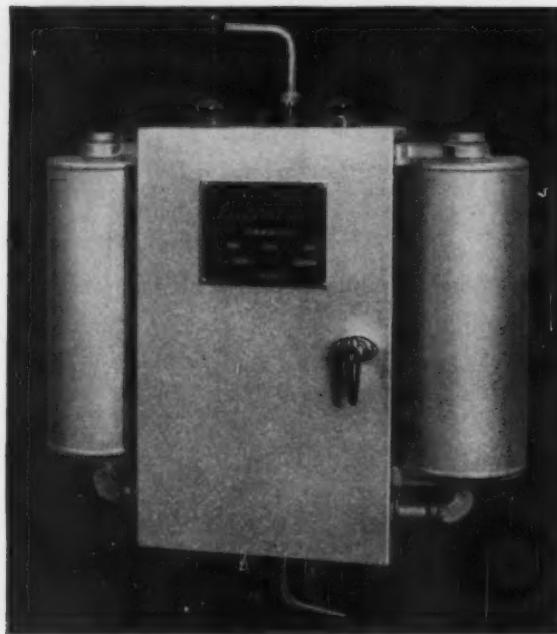
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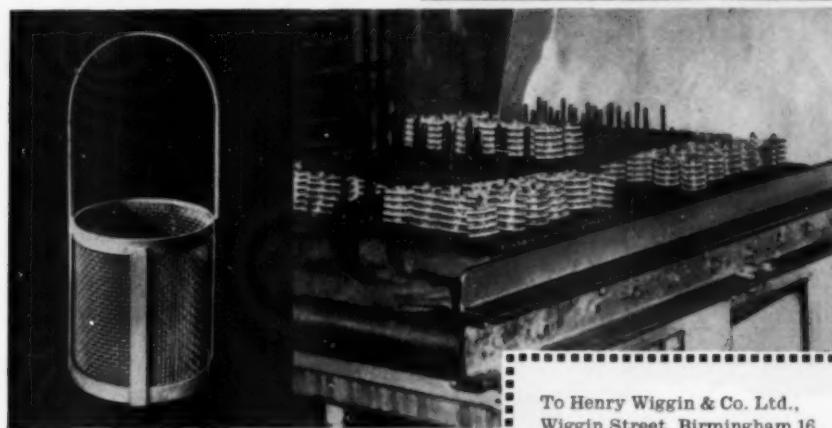
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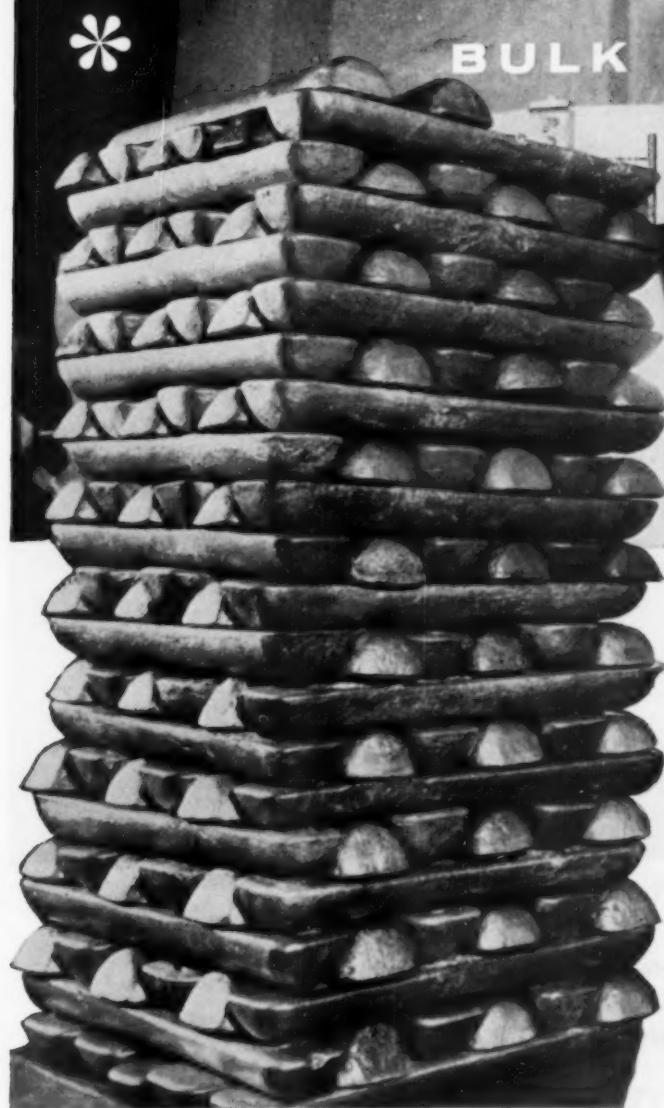
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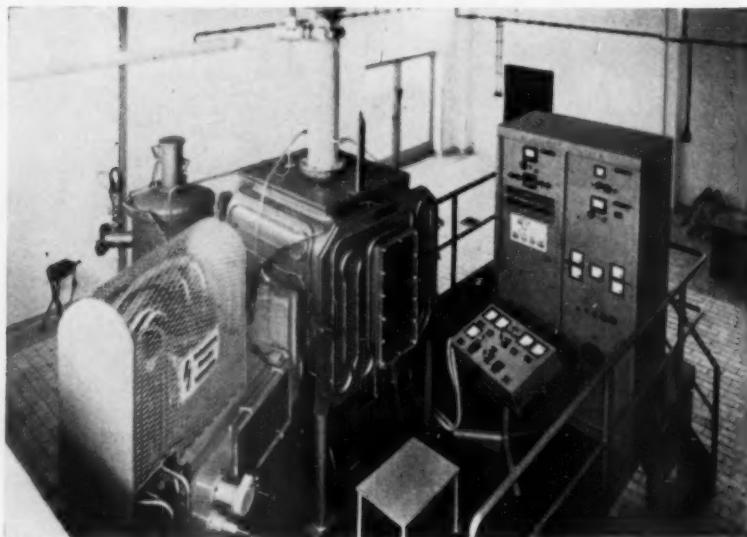


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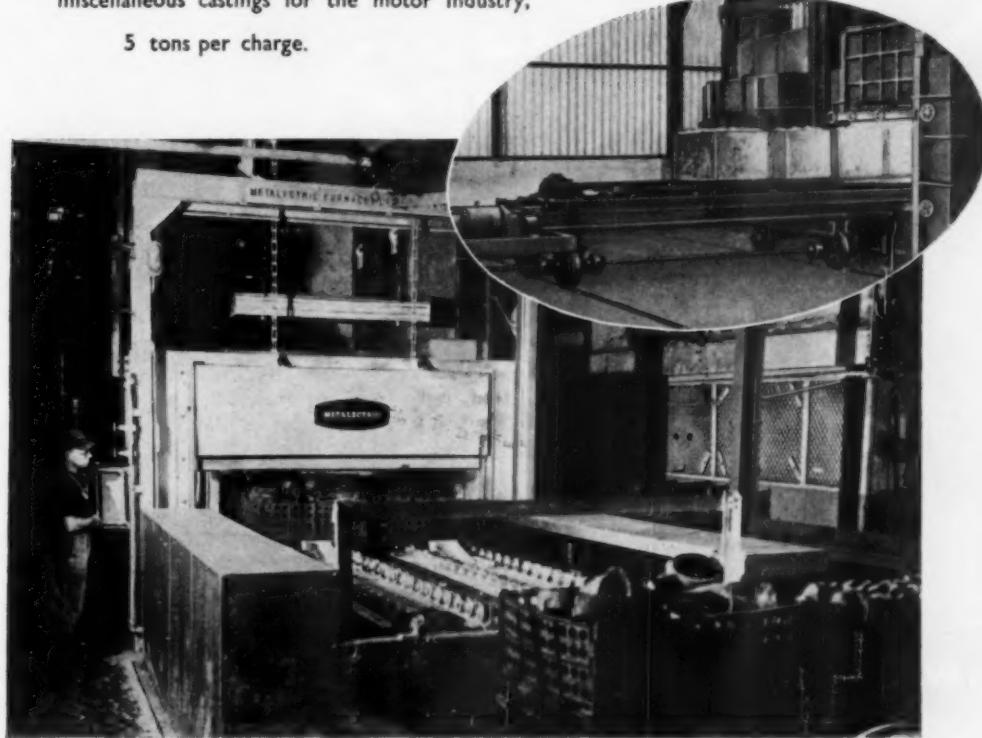
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INCORPORATING THE METALLURGICAL ENGINEER

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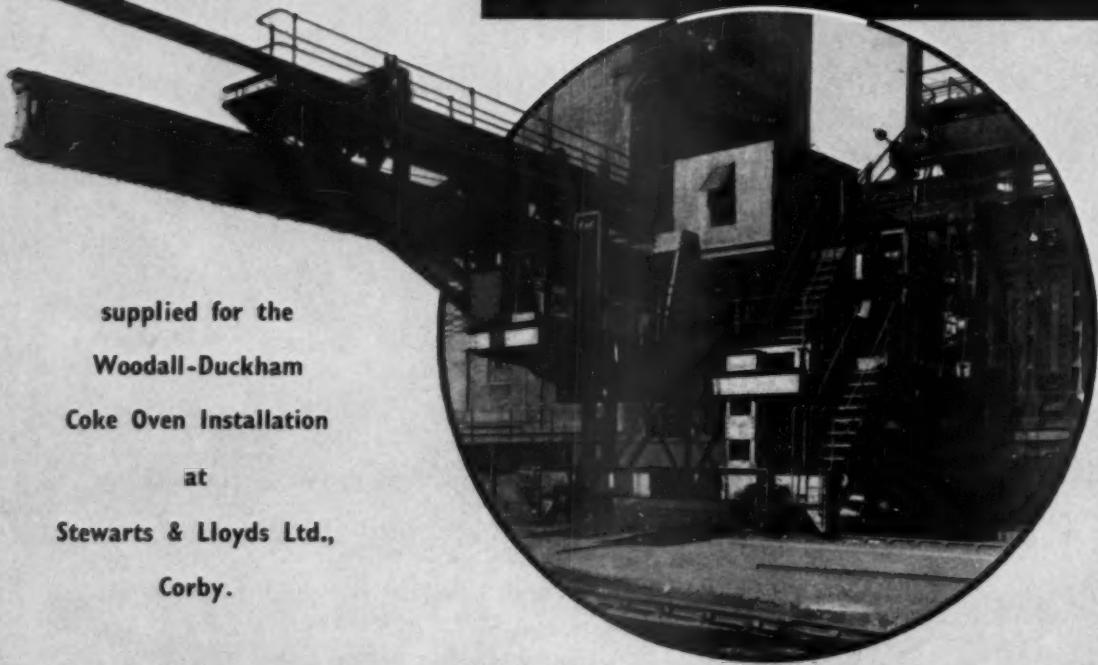
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# METALLURGIA

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JULY, 1961

Vol. LXIV. No. 381

## Automation and Exports

SINCE the war ended the vocabulary of the man in the street has been enlarged by the addition of—among others—the word “automation,” and not unnaturally he has taken this to be something quite new. This is far from being the case, it being no more than an extension of the substitution of mechanical for manual operation which has been going on since industrialisation began. True it now applies to design, transfer, control and inspection procedures and will finally do so to the whole process of manufacture, using electronic controls, computers and other modern devices. Moreover, unlike the original industrialisation, automation extends to the office just as much as to the factory. But, as Lord Hailsham pointed out in his speech at the opening of the recent “Automation—Men and Money” conference at Harrogate: “The essence of the matter is not the substitution of machinery for skill, it is the development of more skill in the use of machinery. The end will be, not less opportunities for skilled employment, but more; not a threat to labour, but an enhancement of its status. This, oddly enough, is the result of all labour-saving devices. To an alert and forward-looking industry, automation should involve headaches, but no heartaches—either for management or for labour.” Although the man on the shop floor has no cause for falling into the Luddite error of thinking that automation must lead to general unemployment, his feeling that it will result in changes in the pattern of employment is amply justified, but such changes must be considered in relation to the national good.

We are at the moment facing yet another economic crisis with the prospect of new forms of “squeeze” to restrict home consumption and encourage exports. The exhortation to “export or bust” has been used so often that many regard it as a political device rather than the economic truth that it is. We cannot feed and clothe a population of 51 millions on home-grown produce and materials, let alone furnish our homes with washing machines, refrigerators and T.V. sets. We must import both food and raw materials, and to pay for them we must export some at least, of the fruits of our labours, for our exportable mineral wealth is almost negligible.

To sell in overseas markets our goods must be competitive. The world doesn't feel that it owes us a living: we cannot live forever on the strength of having stood alone against the might of Hitler's armies in 1940. What we have to sell, therefore, must be not only what the customer wants, but at a price he is willing to pay. How is this to be achieved? We can no longer count on the economic reliability of our technical skills. There is no hope of our retaining competitive power against people with a lower standard of life who are already mastering many of the simplest techniques, and some of

the more advanced ones too, of mass production. We shall be priced out of the market in every case where lower wages, and comparable industrial skill can be combined against us. The textile industry has already felt the “wind of change” in the form of the growing textile industries of Hong Kong, India and Pakistan. The developments in Asian shipyards, too, have given our own shipbuilding industry something to think about. Quotas and import duties have been put forward as a solution to the problem, but in Lord Hailsham's view they will give nothing more than temporary respite, and if unaccompanied by vigorous measures for the longer term they will encourage only moral laziness and complacency.

Lord Hailsham continued: “This competition is at once legitimate and intrinsically healthy. The future of this country, its skill and wealth, is not to depend on keeping others ignorant and poor. Our future depends on seizing and remaining in the lead in industrial design, technology, production and salesmanship. This is not simply a question of workmanship, enterprise and skill. It is above all a question of brains and forethought. We must exploit and develop those advantages that we have. Greater scientific knowledge is one such advantage; a higher level of general and technical education is a second; an already sophisticated standard of engineering is a third. Our aim should be to remain at least one move ahead at every stage, so that, by the time the new countries are beginning to acquire an existing technique, we ourselves shall be moving over rapidly to another. From this point of view automation is a necessity of our national future.”

The wider aspects of automation have been discussed with both sides of industry, through the National Joint Advisory Council, but it is important that the nature of the changes involved should be well and humanely explained to all who are in any way concerned in the introduction of automation in a particular case. Automation is not necessarily the answer to any and every problem of economic production—in some cases traditional methods are still the best—but where, after careful consideration of all the facts, automation is decided upon, the changes involved will call for the highest degree of co-operation between management and unions if the venture is not to be fighting an uphill battle from the start. Relations between management and unions will be intensely affected in the transition stage when the implications of change are discussed in detail. From these discussions new wage structures will emerge and new conditions of work will be established, which may involve different shift systems, different stresses, different dangers and different human relationships.

The introduction of automation in any concern will have far-reaching consequences. Not only will the skill of the operator be replaced by the skill of the maintenance technician, but there will be different management skills

required, and even the nature of managements' authority may alter. Again, the capital cost involved is such that even longer production runs are necessary to make a profit. Thus the salesman and advertiser are heavily involved and the provider of risk capital must wait

### Expanding Use of Galvanizing

OVER 1,200,000 metric tons of zinc (40% of the total world consumption) were used for galvanizing in 1960 according to reports by delegates from all over the world at the 6th International Galvanizing Conference at Interlaken last month. About 6% of the world's steel production is estimated to be hot galvanized to prevent it from rusting. In 1960 the European industry for the first time consumed as much zinc for galvanizing as the United States—approx. 320,000 tons. In the United States the galvanizing of sheet and strip took over half, and nearly three times as much as general galvanizing. In Europe, only Belgium and France used more zinc for sheet and strip than for general galvanizing which is the largest use in most European countries.

A major development in Europe is the increase in the galvanizing of structural steel, much of it by new specially built plants. Railway electrification schemes and power transmission lines are now big users of galvanizing in most countries and are setting a pattern which building and other industries are beginning to follow. From the United States came news of the widespread adoption of galvanized strip by the automobile industries for body construction, especially for underbody panels. Everywhere the use of galvanized strip is expanding, and a further substantial rise in the consumption of zinc for galvanizing is forecast for this year, when new strip galvanizing lines under construction in Europe and other countries begin production.

### European Non-Ferrous Trends

THE activity in non-ferrous metals industries in Europe as a whole should remain in the near future at a level at least as high as that reached by the end of last year, states the Non-Ferrous Metals Committee of O.E.E.C. in its latest brief market survey.\* There are indications that the upward trend of activity may have lost some of the previous year's impetus, but domestic demand for metals and semi-fabricated products, particularly for engineering industries and building activity, justifies the conclusion that the outlook for the near future is satisfactory.

In all member countries, metal consumption went up—in some cases quite spectacularly—last year, and has remained high so far in 1961. Increases in production did not match consumption; imports, and hence dependence on raw material supply from outside, were therefore considerably higher than in previous years. It does not, however, seem likely that 1961 will show another record level. Metal imports from Eastern countries were, on the whole, lower than in previous years. The overall supply situation is expected to remain satisfactory. With regard to the present over-supply of lead in the Western world, the figures show that in O.E.E.C. member countries producer stocks have stayed at a normal level.

\* "Non-Ferrous Metals: Recent Developments and Short-Term Prospects (October 1960—June 1961)." Copies may be obtained free of charge from the O.E.E.C. Information Division, Chateau de la Muette, 2 rue André-Pascal, Paris XVI.

longer between the investment of his money and a return on it. New mass markets are eagerly being sought, and according to Lord Hailsham, current conversations about the European Economic Community have more than an echo of automation in their content.

Processing industries for aluminium and copper continued to work at full capacity; manpower difficulties and consequent long delivery periods for metal products are again reported from a number of countries. The various branches of zinc processing expanded further, although more slowly than in recent years. Demand for lead products made a better show than had been expected in October last year, and demand for nickel remained strong due to the rising production of nickel-alloyed steel.

The main features of the situation in the various member countries, production expansion projects and trends in processing industries, are briefly outlined in the Report.

### Iron and Steel Prices

LAST month the Iron and Steel Board announced its decision to increase the maximum prices of iron and steel products permitted to be charged in the United Kingdom by 1% on all items. The increase was described by the British Iron and Steel Federation as totally inadequate having regard to the recent additional cost burdens on the industry. The Federation estimated the consequential increase in the industry's earnings at a rate of £7.5 million a year over the second half of 1961, as compared with estimated additional costs over the same period at a rate of about £25 million a year net, after taking into account all expected savings from greater efficiencies.

### News in Brief

*Noral News*, the quarterly magazine of Alcan Industries, Ltd., has been awarded a Certificate of Merit "for high quality of content and presentation" in the 1961 National House Journal Competition sponsored by the British Association of Industrial Editors.

CHARLES CLIFFORD, LTD., Dogpool Mills, Birmingham, 30, have been awarded contracts for the supply of zinc wire for metal spraying the surfaces of the fabricated steelwork to be used in the new Forth road bridge, at present under construction.

THE address of the National Council for Technological Awards is now 24, Park Crescent, London, W.1 (Telephone: LANgham 4879).

THE APPLEBY-FRODINGHAM STEEL CO., branch of The United Steel Cos., Ltd., have now put into production their new universal beam mill, engineered by Schloemann A.G., Düsseldorf. The mill is capable of rolling universal wide flange beams with parallel flanges up to 24 in.  $\times$  9 in., universal columns up to 12 in.  $\times$  12 in., and corresponding junior beams.

THE name of the company formerly known as Radio Heaters, Ltd., has been changed to Radyne, Ltd., in line with the company's trade-mark.

# Relaxation Testing of Steam Turbine Bolt Materials with Simulated Re-tightening

By J. H. M. Draper, B.Sc.Tech., A.M.I.Mech.E.

*Research Department, Associated Electrical Industries (Manchester), Ltd.*

*A procedure is described for assessing the probable behaviour of steam turbine cylinder bolts when they are re-tightened a number of times in service. Using this procedure, a 1% Cr-Mo-V bolt steel and a 12% Cr-Mo-V bolt steel have been tested, the results indicating that when bolts in either material are tightened six times the residual stress at the end of any operating period is never less than that at the end of the first. The tests also suggest that different temperatures give the same pattern as regards the creep relaxation in successive operating periods. In such circumstances it is suggested that single loading creep relaxation data can be used for design.*

**A** STEAM turbine cylinder bolt is initially tightened so that some known strain (or corresponding stress) is imposed. During its service life at high temperature the stress, and therefore the load in the bolt, is reduced by creep relaxation. In service the stress remaining in the bolt, i.e. the residual stress, must not fall below a minimum permissible value derived from design considerations regarding the pressure between the cylinder flanges needed to keep the joint tight, and the maximum area of bolt cross-section which it is feasible to provide.

A good deal of creep relaxation testing has already been carried out on bolt materials under conditions which simulate one tightening, and methods of test<sup>1,2</sup> and data<sup>3</sup> obtained from such tests have been reported. In practice, however, bolts would usually be refitted after each turbine overhaul and would therefore be re-tightened several times during their service life. The late Dr. R. W. Bailey pointed out some years ago that creep relaxation behaviour is not necessarily the same in each operating period between tightenings, and can only be assessed by re-tightening tests.

Tests have since been carried out by the N.P.L., by some of the steelmakers, and by ourselves, and valuable results have been obtained, but no standard test procedure has been followed, so that direct comparison of results is not possible. Some comments are made below on the different procedures followed, and a revised procedure is proposed which it is hoped might form the basis of a standard procedure throughout the turbine industry. Unpublished proposals on these lines have already been made by the author at a discussion on re-loading techniques at the N.P.L. in 1959 and at an International Conference in Düsseldorf in 1960.

## Service Conditions

In practice, turbine cylinder bolts are usually tightened to a strain of 0.15%, although some turbine builders use other strains, generally greater. Under elastic conditions at room temperature this corresponds to a stress of 20 tons/sq. in., but at working temperature the stress will, of course, be lower because (a) the modulus of elasticity is lower, and (b) the strain may be partly plastic. Assuming that the flanges are perfectly rigid, the total strain remains constant during an operating period, but the stress diminishes with time.

Where ferritic bolt materials are used, joints are

usually designed so that the minimum bolt stress required to maintain the joint is between 3 and 5 tons/sq. in.

A power station turbine designed for a life of 20 years, would normally be overhauled at 30,000 hour intervals, so that there would be six operating periods of equal duration and the bolts would accordingly be tightened-up six times.

## Previous Tests

Previous tests by different investigators have differed mainly in the procedure adopted to simulate re-tightening and the duration of the test cycle. Re-tightening has been simulated by one or other of the following methods:

- (a) Applying a total strain (residual elastic + additional strain) equal to the specified strain for the first loading.
- (b) Raising the stress to the maximum value recorded on the first loading.
- (c) Adding a strain of specified magnitude, e.g. 0.1% in a test in which the initial strain is 0.15% and the residual stress in the previous loading is 5 tons/sq. in. (Making the total strain only approximately equal to the initial strain).

Method (a) is the only one which correctly simulates service conditions as regards strain, and is preferred since it is as convenient to use as (b) or (c), which give strains only approximating to the correct value.

The duration of the relaxation periods has been decided in one of the following ways:

- (d) The first re-loading is carried out when the residual stress has fallen to a specified value. Subsequent re-loadings are not made until this same stress is reached again.
- (e) The first re-loading is carried out after the initial stress relaxation has continued for a specified time. Subsequent re-loadings are made at intervals equal to this specified time.

Method (d) permits the adoption of a stress which can be related to the minimum bolt load required in practice, but does not simulate the practical case in that re-tightening of cylinder bolts normally takes place at equal time intervals. Method (e) on the other hand, does simulate the practical time cycle, but the residual stresses obtained in the test are not necessarily related to those required by service conditions. Incidentally, method (e) is more convenient in the laboratory as it

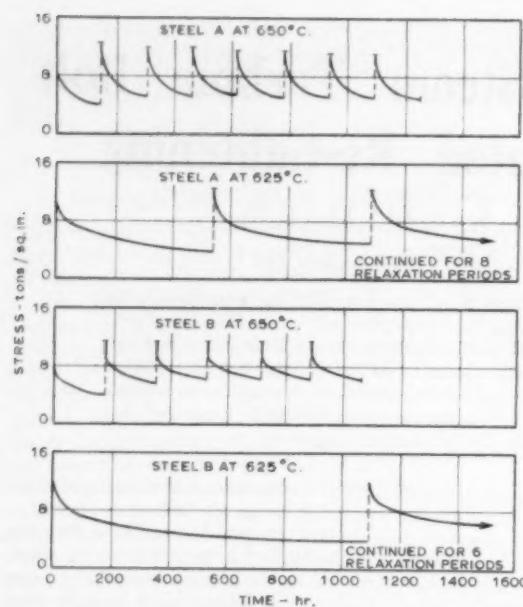


Fig. 1.—Stress-time relationships for creep relaxation tests with re-loading at 650° C. and 625° C.

facilitates the programming of work and reduces the total testing time. The procedure now proposed adopts method (a) together with what is felt to be the best compromise between (d) and (e).

#### Proposed Procedure

In the proposed procedure it is suggested that a test specimen should be subjected to a first relaxation period with a specified strain and temperature as in a normal relaxation test. When the residual stress has fallen to a specified value the specimen is re-loaded while still at test temperature. Subsequent re-loadings are made at intervals of time which are equal in duration to the first relaxation period. Test conditions would be selected in accordance with the following considerations.

#### Temperature of Tests

For each test the temperature can be chosen to shorten the time for relaxation to the predetermined residual stress—and therefore the time between subsequent re-loadings—to some convenient value, but it should not be shortened so much as to make the temperature unduly high. If a normal series of creep relaxation tests at different temperatures has already been carried out on the material, the choice of a suitable test temperature is easy.

#### Strain in First Relaxation Period

A strain of 0.15% is proposed as this appears to be acceptable to most concerns. Other strains would be used, however, if it were thought that they might be adopted in practice.

#### Residual Stress at First Re-loading

The stress at which re-loading is first carried out in a test should ideally be the same as the stress remaining in the turbine bolts just before the first overhaul. With a specified initial strain and a specified time to the first

overhaul, this stress will depend on the service temperature, and it is obviously not possible in a single test to simulate the conditions obtaining at different service temperatures. It seems reasonable, however, to simulate the most severe conditions practicable in service in which the residual stress in the bolts falls to the minimum permissible value needed to keep the joints tight. For normal ferritic steels this value is usually of the order of 3-5 tons/sq. in., and it is therefore suggested that the first re-loading in re-tightening tests should be carried out when the residual stress has fallen to 4 tons/sq. in. With more advanced materials of superior creep properties, however, this minimum permissible stress may have a different value.

#### Strain after Re-loading

It is proposed that re-loading should be carried out with the test specimen still at test temperature, and that strain should be increased by an amount which, when added to the elastic strain calculated from the residual stress at the end of the previous relaxation period, gives a total strain of 0.15%.

In practice, at overhaul times, bolts cool-off with the load still on and when removed have a new length due to the creep strain which has occurred. On re-assembly a strain of 0.15% is added to the new length before the temperature is re-applied. It is not possible to copy this cycle with a normal creep machine, where strain can only be measured at a constant temperature because extensometer design is such that strain and thermal expansion cannot be separated during heating and cooling. The proposed procedure is considered reasonable, however, in view of the fact that tests<sup>2,5</sup> have been made which indicate that the results will be independent of the straining procedure except in the very early stages.

The only alternatives to this procedure appear to be:—

(i) To remove the load when cold and re-load at test temperature. This is considered undesirable since it would entail the specimen being at temperature without load. Under these conditions some creep recovery would take place and the subsequent relaxation would be affected.

(ii) Construction of special creep relaxation machines in which the strain recording is uninfluenced by changing temperature and in which, therefore, the strain can be applied cold, and maintained whilst heating. Examples of such machines have been described by Bihet, Gottignies and Caubo<sup>4</sup> and by Watson.<sup>5</sup>

#### Number of Re-loadings

It is proposed that the test should consist of six loadings, i.e. five re-loadings in accordance with the service conditions mentioned above.

#### PROCEDURE AND CONDITIONS FOR FERRITIC BOLT MATERIALS

Summarising the above, the proposed procedure for ferritic steel bolt materials is as follows:—

#### First Loading

(i) With the test specimen at test temperature, a strain of 0.15% is applied.

(ii) Relaxation is allowed to take place until the residual stress has fallen to 4 tons/sq. in.

### Subsequent Re-loadings

- (iii) With the test specimen still at test temperature, strain is increased by an amount which, when added to the elastic strain calculated from the residual stress at the end of the previous relaxation period, gives a total strain of 0.15%.
- (iv) Relaxation is allowed to take place for a length of time equal to that occupied by the first relaxation period.
- (v) This procedure is repeated until six relaxation periods have been completed.

### Application of Results to Service Conditions

Re-tightening tests that have been made so far indicate differences in the creep behaviour in various relaxation periods. Although in most cases there is some improvement after the first loading, it seems unlikely that any advantage could be taken of this in design. In these circumstances, the purpose of the tests should be to show whether or not there is any deterioration as a result of the six successive loadings.

In the absence of such deterioration the maximum practicable working temperature will be that at which the residual stress just falls to the minimum specified value at the time of the first overhaul, say after the first 30,000 hours of service. This limiting temperature can be derived by extrapolation from single loading creep relaxation tests.

If deterioration is revealed, the maximum working temperature derived from single loading tests will be too high for the specified minimum residual stress. If the deterioration is severe, as in the case of an experimental 12% Cr-Mo-V steel tested some years ago by the author, the material will probably be rejected, unless it possesses some special merit which more than compensates for its poor re-tightening characteristics.

Where, however, re-tightening data is required on a material which deteriorates, one of the following procedures should be adopted :—

(a) If the lowest stress recorded is too low for reasonable design, further tests will be required, following the suggested procedure but with the residual stress at the end of the first relaxation period increased to give a minimum residual stress during six relaxation periods reasonable for design. A number of tests may be required by this method before the desired result is achieved, but the selection of design stresses will be difficult whatever re-tightening test procedure is adopted.

(b) Where the lowest stress recorded, although less than the specified minimum stress for the test, is reasonable for design, no further testing will be required.

In either case, the residual stress at the end of the first relaxation period will be used to estimate the

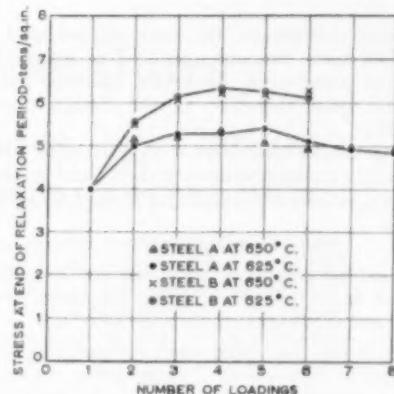


Fig. 2.—Effect of re-loading on residual stresses at 650°C. and 625°C.

maximum working temperature for a 30,000 hour life from single loading data, and the minimum residual stress will be used for design.

### Re-tightening Tests on Two Bolt Steels

Re-tightening tests have been carried out with the dual purpose of checking the proposed procedure and of assessing the re-tightening properties of two ferritic bolt steels :—

*Steel A* A 1% Cr-Mo-V steel which is already commonly adopted for bolts for high temperature steam turbines, and

*Steel B* A 12% Cr-Mo-V steel which shows promise as an alternative material.

Tests at temperatures of 650°C. and 625°C. have been completed and further tests are now in progress at 600°C. Although, as has been pointed out, six relaxation periods are sufficient, as a matter of interest tests on Steel A were continued to a total of eight relaxation periods. The results are shown in Table I and the relaxation curves are reproduced in Fig. 1. Fig. 2 shows the residual stress at the end of each relaxation period plotted against number of loadings at each of the test temperatures for the two steels. The results show that :

(a) At no time, in any of the tests, was the residual stress less than that at the end of the first relaxation period.

(b) Considering each material separately, at both the test temperatures used, the residual stresses at the ends of corresponding relaxation periods were approximately the same.

The additional tests which are in progress at 600°C. are expected to confirm these features at a third temperature. Although such further evidence is necessary to

TABLE I.—RESULTS OF TESTS ON 1% Cr-Mo-V AND 12% Cr-Mo-V BOLT STEELS

| Material | Temperature (°C.) | Time for Stress to Fall to 4 tons/sq. in. in First Loading (hr.) | Residual Stresses at the Ends of the Indicated Relaxation Periods (tons/sq. in.) |      |      |      |      |      |      |      |
|----------|-------------------|--|--|------|------|------|------|------|------|------|
|          |                   |  | 1st  | 2nd  | 3rd  | 4th  | 5th  | 6th  | 7th  | 8th  |
| Steel A  | 650               | 157  | 4  | 5.18 | 5.19 | 5.33 | 5.03 | 4.91 | 4.95 | 4.89 |
|          | 625               | 546  | 4  | 4.97 | 5.26 | 5.29 | 5.40 | 5.08 | 4.92 | 4.78 |
| Steel B  | 650               | 178  | 4  | 5.20 | 6.05 | 6.21 | 6.21 | 6.25 | 6.14 | —    |
|          | 625               | 1095   | 4  | 5.54 | 6.12 | 6.33 | 6.25 | 6.14 | —    | —    |

have complete confidence, the tests suggest that these materials will have the same pattern as regards creep relaxation in successive relaxation periods at other temperatures, and therefore at the design operating temperature.

With both steels, therefore, design of bolts could be based on single loading relaxation data, and such bolts would remain satisfactorily tight for at least six operating periods.

#### Conclusions

(1) A revised procedure is proposed for re-tightening tests on bolt materials which closely simulates practical conditions and is convenient for laboratory use.

(2) The procedure has been used successfully for tests on two ferritic bolt steels. These tests showed that successive re-tightening resulted in some improvement of the relaxation properties of the two materials concerned.

(3) Whenever an assurance can be obtained that there will be no deterioration of relaxation properties as a result of re-tightening, single loading relaxation data can be used for design purposes.

#### Acknowledgments

The author wishes to thank Sir Willis Jackson, F.R.S., Director of Research and Education, Associated Electrical Industries (Manchester), Ltd., for permission to publish.

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### G. A. Harvey & Co. (London), Ltd.

HARVEY'S new head sales office and showroom in Villiers House, Strand, W.C.2., was officially opened on Monday, 26th June by Mr. Paul Reilly, Director of the Council of Industrial Design. The new office and showroom are on the first floor of a modern office building and have an area of 6,500 sq. ft. In the showroom are exhibited many items from the Harvey and Harvey-Milner ranges of steel office furniture and partitioning, while in the reception area are coloured transparencies showing examples of production from Harvey's other departments which include heavy fabrication, light fabrication, sheet metalwork, metal perforation, wirework and weaving and galvanising. The offices provide accommodation for senior personnel of the sales department and also for the manager, sales representatives and staff of the London branch office.

New branch offices and showrooms of similar style and each of about 1,200 sq. ft. in area have recently been opened in new office buildings in Bristol, Leeds and Glasgow and three more will be opened in July in

Manchester, Liverpool and Birmingham. All these provincial offices are linked with Villiers House and with the works at Greenwich by Telex communication.

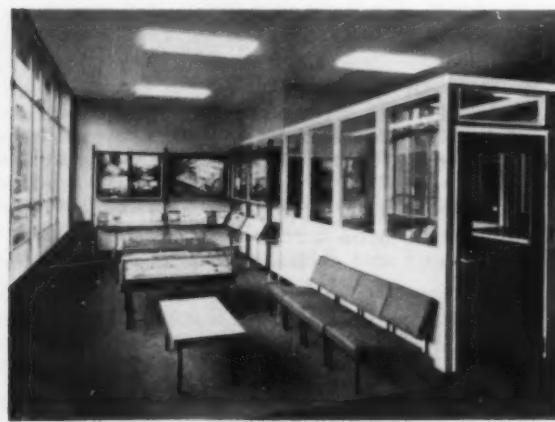
The opening of the new head sales office and showroom in Villiers House coupled with the chain of six new provincial offices provides a notable milestone in the history of the company. From the humble beginnings of an old smithy shop in Lewisham in 1874, where Mr. George Alfred Harvey founded the firm with the help of one boy, the works, removed to Greenwich in 1913, now occupies an area of 28 acres. There are a further 2 acres at the Margate factory and in all Harveys now have over 2,600 employees.

### Powder Metal Spray Pistol

FOORTY-TWO representatives from twenty-three North Eastern companies recently watched a demonstration of the new Berk 61 metal spraying pistol at the Middlesbrough works of Dorman Long (Bridge and Engineering) Co., Ltd. The demonstration was organised by the coating division of F. W. Berk & Co., Ltd., who have developed the Model 61 pistol to meet the requirements of contractors who need fast spraying equipment, either to cover large areas of structural steelwork, or to match in with automatic grit-blasting installations.

The Berk 61 pistol will deposit zinc powder with a deposition efficiency as high as that obtained with a 35 p.p.h. pistol, with the same gas and air consumption, but at a rate of 110 lb./hr. This is approximately equivalent to the deposition of a 0.004 in. coating over 650 sq. ft./hr. This performance is achieved by means of the patented nozzle, which projects powder, gas and air through alternate and concentric jets. This nozzle, which can be cheaply and easily replaced, is the only part of the pistol which can require maintenance. As the pistol is intended for large scale spraying, it is correspondingly light, well balanced and easy to handle.

Other equipment shown at the demonstration included the Berk Model 50 pistol, used as a pacemaker to the Model 61, the Berk 58 wire gun, and Berk grit-blasting units. A new development is the "Slim Jim" grit-blasting machine which has been designed for lowering through small (18 in.) manholes where the internal surfaces of tanks are to be blasted.



Enquiry office, Telex centre and reception area in the new head sales office and showroom of G. A. Harvey & Co. (London), Ltd., in Villiers House, Strand, London, W.C.2.

# Low Pressure Metallurgy

## The Reduction of Oxides by Carbon

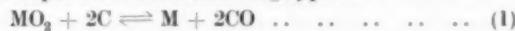
By A. S. Darling, B.Sc.(Eng.), Ph.D., A.M.I.Mech.E.

*This is the third article by Dr. Darling under the general heading "Low Pressure Metallurgy" and the reference numbers follow on those in the earlier articles, the first of which took the form of a general survey, the second dealing with vacuum as an inert atmosphere. In discussing the reduction of oxides by carbon at low pressure, the author considers in this first instalment the factors affecting the progress of reduction, including metal-crucible reactions, before dealing next month with specific examples in the case of copper, molybdenum, chromium, ferrochromium and niobium.*

### The Stability of Carbon Monoxide

THE change of entropy associated with the formation of carbon monoxide from its components is positive, so that the free energy change of the reaction increases with rise of temperature. For this reason the stability of carbon monoxide does not decrease so rapidly with temperature as those of metallic oxides, all of which can be reduced by carbon at sufficiently elevated temperatures. The reduction process leads to an increase in volume and is therefore favoured by a reduction of pressure. This principle is utilised in vacuum metallurgy to ensure complete deoxidation.

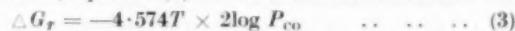
The reduction process is conveniently represented by an expression of the following type:—



The equilibrium constant of this reaction is related to its free energy by equation (2)



Because carbon monoxide is the only uncondensed phase present, equation (2) can be rewritten in the form:—



This expression relates the free energy of the reaction (the reactants being initially considered in their standard states) to the position of equilibrium finally achieved when the free energy is zero. The pressure of carbon monoxide in equilibrium with the pure metal and metallic oxide is expressed in atmospheres.

The free energy change can be considered as a measure of the mechanical work done during the reaction. The work is that done by the isothermal expansion of 2 gram molecules of carbon monoxide from atmospheric down to the equilibrium pressure and is therefore indicative of the

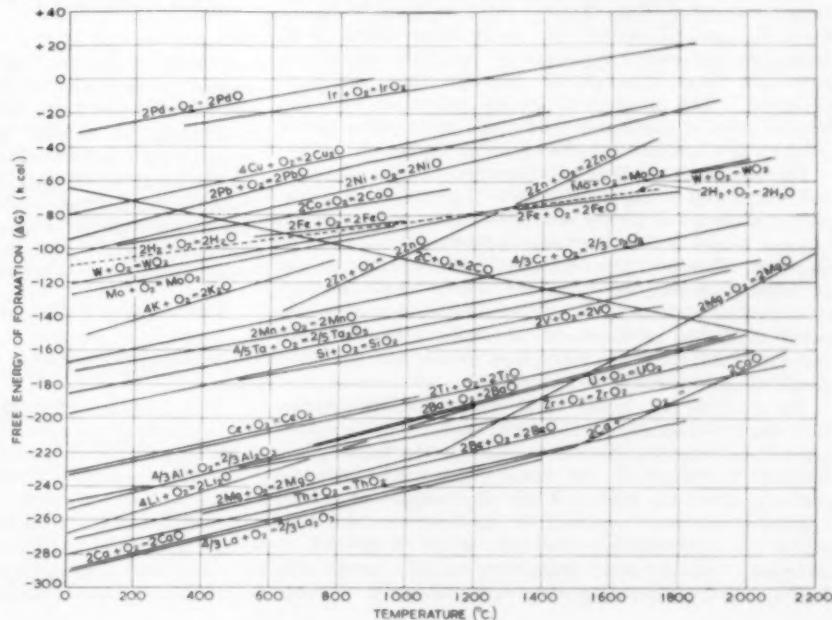
Fig. 1.—Free energy of formation of metallic oxides and carbon monoxide.

tendency of the metal to reduce carbon monoxide rather than that of carbon to reduce the metallic oxide. The higher the free energy, the greater will be the pressure range over which the gas is capable of expanding and the lower will be the pressure of carbon monoxide in equilibrium with the metal and metallic oxide.

Free energy is an additive quantity and is conveniently obtained by considering the basic components of equation (1):—



Fig. 1 illustrates the conventional method of plotting curves of the free energy of formation of metallic oxides and carbon monoxide. The free energy of the reaction represented by equation (1) is zero when the carbon monoxide line intersects that of the metallic oxide. At this point the metal and its oxide are in equilibrium with carbon monoxide at atmospheric pressure. Reduction in such instances will proceed only if the carbon monoxide



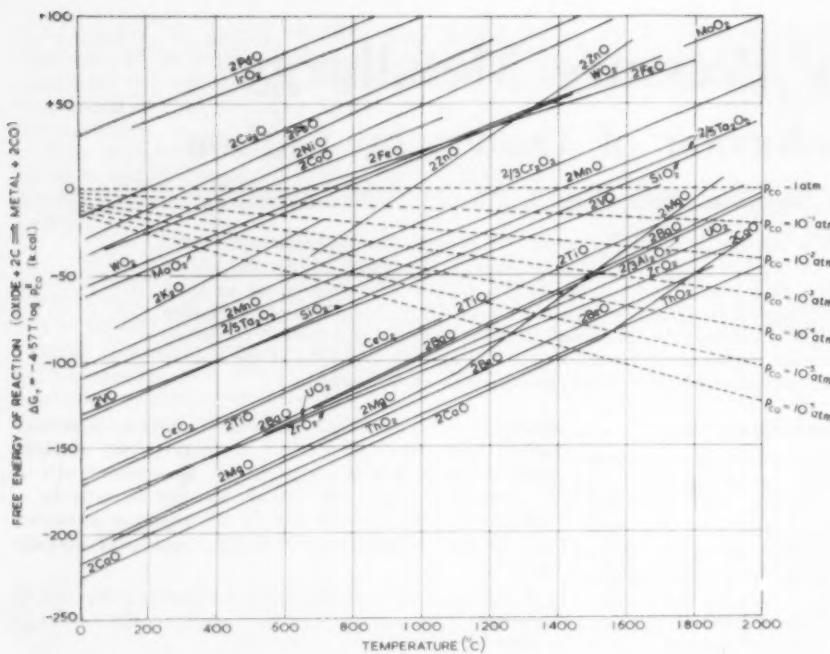


Fig. 2.—Free energy of carbon reduction processes and constructions for deriving equilibrium carbon monoxide pressures.

conditions. Table I, which summarises data from a number of papers dealing with the vacuum fusion process, indicates the completeness of the reduction attainable.

#### Prior Carbide Formation

The affinity of the metal for carbon may be greater than the tendency towards oxide reduction. Examples of this tendency are afforded by titanium and zirconium, both of which form very stable carbides. Although certain titanium-bearing alloys containing carbon are vacuum melted with consequent reduction in oxygen

content,<sup>156</sup> the carbon is sufficiently in excess to permit a certain amount of carbide formation. Sully, Brandes and Provan<sup>157</sup> found that chromium alloys containing appreciable quantities of titanium or zirconium could not be effectively deoxidised with carbon. Kroll<sup>158</sup> reported that zirconium containing a few tenths of 1% of carbon and oxygen, did not evolve carbon monoxide when heated to 2,000° C. in a vacuum of one micron.

The direct reduction of titanium oxide with carbon might be represented as follows:—



The free energy of this reaction at 1,727° C. is 23,100 cal./mole, corresponding to an equilibrium carbon monoxide pressure of 2.2 mm. of mercury. Rapid reduction of this oxide might be expected under vacuum conditions, if it were not for the greater tendency towards carbide formation. We can visualise an attempt to eliminate small quantities of oxide from a titanium melt by stoichiometric additions of carbon. At 1,727° C. the free energy of carbide formation is 48,600 cal./mole. Carbide formation will, therefore precede oxide reduction, the final reaction being as follows:—

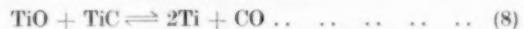


TABLE I.—COMPLETENESS OF REDUCTION ATTAINABLE BY USE OF EXCESS CARBON UNDER HIGH-VACUUM CONDITIONS.

| Oxide                   | Operating Temperature (°C.) | Reduction Achieved (percentage of theoretical) | Reference |
|-------------------------|-----------------------------|--|-----------|
| $\text{Al}_2\text{O}_3$ | 1,000                       | 98   | 152       |
| $\text{SiO}_2$          | 1,550                       | 98.5   | 152       |
| $\text{MnO}$            | 1,550                       | 98.2   | 152       |
| $\text{Mn}_3\text{O}_4$ | 1,580                       | 98.8-100                                       | 153       |
| $\text{Cr}_2\text{O}_3$ |                             | 100  | 153       |
| $\text{ThO}_2$          | 1,900                       | 100  | 154       |
| $\text{TiO}_2$          | 1,800                       | 98-100   | 154       |
| $\text{UO}_2$           | 1,700                       | 100  | 154       |
| $\text{V}_2\text{O}_5$  | 1,540                       | 100  | 154       |
| $\text{ZrO}_2$          | 1,625-1,750                 | 98-100   | 154       |

is continually extracted so that its pressure never exceeds atmospheric.

In Fig. 2 the difference in free energy of reactions (4) and (5) has been plotted against temperature so that the free energy of reaction (1) can be read off directly. The magnitude of this free energy change is related to the equilibrium pressure of carbon monoxide by equation (3). Positive free energy changes indicate that the reduction can only proceed at pressures below atmospheric.

The dotted lines on Fig. (2) represent the values of the expression  $-4.57T \times 2\log P_{\text{CO}}$  for temperatures up to 2,000° C., and illustrate the mechanical work done during the isothermal expansion of two moles of carbon monoxide from atmospheric pressure down to that indicated. The pressure of carbon monoxide in equilibrium with a particular oxide and metal at a given temperature can be read directly from the graph.

The data in Fig. 2 illustrate, for example, that alumina will be reduced by carbon at 1,600° C. providing that the partial pressure of carbon monoxide in the reaction zone is kept below  $3.3 \times 10^{-3}$  atmospheres or 2.5 mm. of mercury. Zirconia, a more stable oxide, is only reduced at carbon monoxide pressures below 0.38 mm. of mercury, while the reduction of beryllia requires pressures below 0.08 mm. of mercury. Such data illustrate the relative stability of the refractory oxides and permit a rough assessment of their reducibility.

The analysis has, so far, been based upon the assumption that the oxide, carbon, and metal exist as pure phases, and that equilibrium conditions can be uniquely defined in terms of the carbon monoxide pressure directly above the melt. While the weaknesses of this argument are very apparent, it must be observed that where an appreciable excess of carbon is present in solution, even the most refractory metallic oxides can be rapidly and quantitatively reduced under vacuum

The free energy of this reaction at the same temperature is 71,700 cal./mole, corresponding to an equilibrium carbon monoxide pressure of approximately  $10^{-5}$  mm. of mercury. Under such conditions vacuum reduction would proceed at a very low rate.

If the whole object is to reduce the oxide, and a large excess of free carbon is not a disadvantage, reduction can proceed in spite of carbide formation. In the vacuum fusion method of oxygen determination, rapid and quantitative reduction of titanium dioxide occurs at 1,750°C. Although it might be assumed from the thermodynamic data that the mechanism of reduction would involve the direct formation of carbide, such apparently is not the case. Sloman<sup>154</sup> suggests that the oxide reacts with carbon and with molten iron to form carbon monoxide and a solution of metal in the iron. The experimentally determined rate of reaction suggested an equilibrium carbon monoxide pressure of about 2.4 mm. of mercury, which was in agreement with the value calculated from the thermodynamic data of the reduction solution mechanism.

Complete reduction of the oxide to titanium by carbon appears to be impossible, however. This was attempted by Kroll in 1948.<sup>155</sup> Finely powdered oxide and graphite were mixed in the correct proportions and briquetted. When heated in a vacuum furnace, reduction commenced at 1,100°C. At 1,800°C. the pressure had decreased to 8 microns. When gas evolution ceased the briquette was cooled and weighed. Complete reduction to the metal would have involved a weight loss of 54%. The observed weight loss was 74%. Kroll interpreted this finding as an indication that the lower oxide of titanium was volatile. The reduction process yielded a product containing 20.7% of carbon and 73.7% of metal, the remaining 5.6% being oxide.

Experiments with zirconium yielded similar results. Oxide-metal, and oxide-carbide mixtures were heated to very high temperatures without appreciable reduction. The carbide-oxide mixture was heated to 1,960°C.—well above the melting point of zirconium—after which the product still contained 7.5% of oxygen and 6.2% of carbon.

It appears that with both titanium and zirconium the affinity for carbon precludes a complete reduction of oxide to metal. Carbide and oxide, or carbide and dissolved oxygen can coexist in these metals. The carbide of vanadium is somewhat less stable, but even with this metal quantitative reductions are impossible. Kroll obtained vanadium containing 0.97% of carbon by heating in vacuum a briquette containing vanadium oxide and carbon powders mixed in the correct stoichiometric proportions. The oxygen content, after fusing the mixture in a beryllia crucible was estimated at 5%. Crucible reaction could have accounted for much of this contamination. The essential fact, however, was that a large excess of oxygen did not completely eliminate carbon during fusion *in vacuo*.

Niobium oxide was not successfully reduced with carbon in this series of tests. The final product, obtained after several sintering and crushing operations, still contained large quantities of carbon and oxygen. After final fusion, at 2,400°C. on a bed of thoria, the product still contained 1.01% of carbon and 19.8% of a material, insoluble in HF, which was presumed to be oxide. It was concluded that a large excess of oxygen had to be present to reduce the carbon content to an acceptable

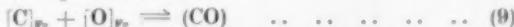
figure. These results are difficult to reconcile with those of recent investigators who have demonstrated the effectiveness of carbon as a deoxidant for niobium. The high oxygen contents are obviously attributable to reaction with the thoria. The carbon discrepancy is partly explicable in terms of the quality of vacuum employed. The lowest pressure reported by Kroll was 2 microns. Williams<sup>157</sup> used a system capable of reducing the partial pressure of carbon monoxide to about  $2 \times 10^{-5}$  mm. of mercury. The ratio of the two operating pressures is virtually the same as the ratio of the two carbon contents obtained. Exact data as to the activity of carbon and oxygen in solid solution in niobium is not available. All the proposed reduction mechanisms postulate independent diffusion of carbon and oxygen to free surfaces before reaction. The solubility of carbon in niobium is very low, however, and it is commonly supposed that the rate of reaction is controlled by this factor. Ductile niobium is produced from raw material having an oxygen content of the order of 0.5%. Oxygen levels of the type encountered by Kroll might effectively prevent interstitial solution of carbon in the niobium lattice, thereby preventing efficient operation of the reduction mechanism.

### Volatile Metals

The oxides of volatile metals are not, in general, effectively reduced by carbon *in vacuo*. Back reactions between the metal vapour and evolved carbon monoxide occur during the reduction process. The condensed product is thereby contaminated by oxide and carbon. Heussler<sup>158</sup> found that MnO and SiO<sub>2</sub> when mixed with carbon and heated *in vacuo* yielded a condensate consisting largely of carbide and oxide. Similar results were obtained by Thompson<sup>151</sup> with calcium and barium oxides, by Kjellgren<sup>152</sup> with beryllium oxide, and by Watts<sup>153</sup> with magnesia.

### Mechanism of the Reduction Process

Carbon and oxygen are generally present in the form of dilute solutions in the melt or reacting body. The basic reduction process can be represented by an expression similar to equation (9), where the solvent is assumed to be liquid iron:



It might be concluded that for any given pressure of carbon monoxide the product of carbon and oxygen concentrations would be constant. The presence of carbon reduces considerably, however, the activity of oxygen in liquid iron, leading to an increase in the value of the equilibrium carbon-oxygen product with rise in carbon content.<sup>155</sup> The carbon-oxygen product under equilibrium conditions varies with carbon monoxide pressure in a perfectly linear fashion, although this behaviour is often obscured at pressures in the vicinity of atmospheric by the appreciable solubility of carbon monoxide in the liquid iron.<sup>156</sup>

Under vacuum conditions, however, such dissolved gas is readily evolved. Fig. 3, largely extrapolated from Chipmans data, agrees with similar information published by J. H. Moore.<sup>159</sup> The curves predict the effect of gas pressure upon the carbon-oxygen relationship in liquid iron at 1,600°C. For purposes of comparison, curves showing the efficiency of various standard metallic deoxidants have been appended to this illustration. By

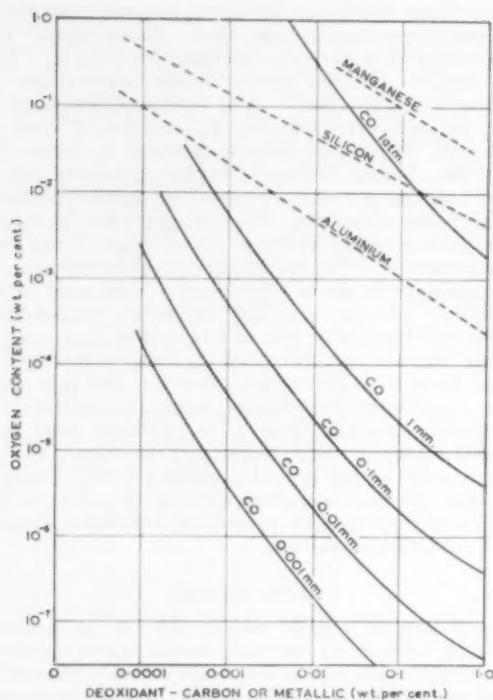


Fig. 3.—Equilibrium carbon oxygen contents in liquid iron at 1,600°C. (From data by Chipman<sup>168</sup> and Moore.<sup>169</sup>)

employing low melting pressures, very low carbon oxygen values become attainable. At pressures of the order of 1 mm. of mercury, for example, carbon becomes a more efficient deoxidant than aluminium. Tix<sup>128</sup> has commented in this connection that vacuum remelting can, by breaking down the alumina, reduce considerably the oxygen content of fully killed steel.

Fig. 4, which illustrates some results obtained by

investigators at the Ford Motor Company,<sup>160</sup> compares the effectiveness of carbon and hydrogen as deoxidants for iron. The curves relate to 14 lb. induction melts of electrolytic iron deoxidised with either hydrogen or with 0.1% of carbon. The hydrogen was dried to a dew point of  $-45^{\circ}\text{F}$ . before circulation over the molten metal surface. The carbon-oxygen reduction process was carried out at pressures in the range 1-12 microns. The rapid rate of the carbon reduction is particularly evident. The authors reported with both processes a practicable oxygen limit of 0.001%. This value was attained with carbon in less than half an hour, whereas four hours of the hydrogen treatment only reduced the oxygen content to 0.0015%.

#### Rate of Reaction

The data in Fig. 3 have been derived largely from the results of experiments made under equilibrium conditions, and it is pertinent at this stage to enquire whether comparable values are obtained during normal vacuum melting operations. The partial pressure of carbon monoxide over the melt is usually indeterminate, and similar in many instances to the vapour pressure of the metal. Because of hydrostatic pressure, vacuum conditions can only exist at the extreme surface of the melt where all the reduction processes must occur. The state of turbulence in the melt will influence considerably the rate of reaction. Metal-crucible reactions may also impose a limit below which the oxygen content of the bath cannot be reduced.

Effects such as these were studied by Thomas and Moreau, who in 1946<sup>161</sup> remelted, under low pressure conditions in magnesia crucibles, steels containing known quantities of carbon and oxygen. The use of a high frequency furnace ensured efficient stirring of the melt. The furnace atmosphere, which contained an indeterminate quantity of carbon monoxide derived from the melt, was maintained at the desired total pressure level by additions of pure nitrogen. After remelting for various periods the melts were rapidly solidified, and oxygen determinations made by vacuum fusion methods. Figs. 5 and 6, which summarise some of the experimental

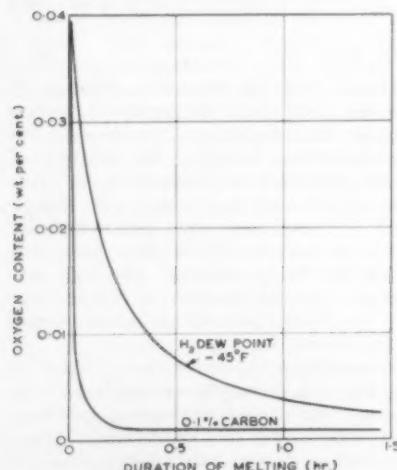


Fig. 4.—Comparison of carbon and hydrogen as deoxidants for molten electrolytic iron. (Morgan and Frey.<sup>160</sup>)

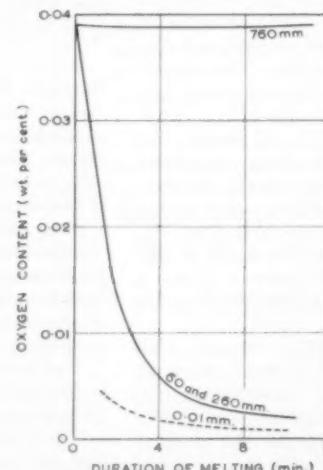


Fig. 5.—Reduction of carbon and oxygen contents during the vacuum remelting of steel. (Thomas and Moreau.<sup>161</sup>)

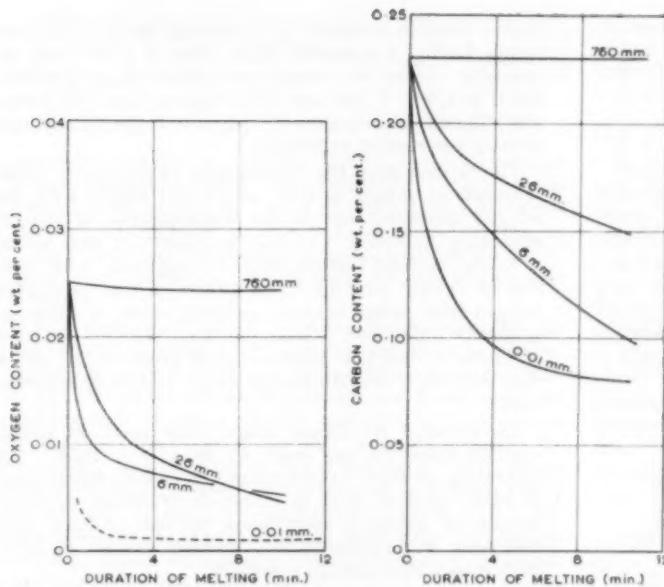


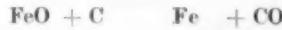
Fig. 6.—Reduction of carbon and oxygen contents during the vacuum remelting of steel. (Thomas and Moreau.<sup>161</sup>)

results obtained, illustrate the effect of time and total pressure on the carbon-oxygen relationship.

No reduction occurred when melting was carried out at atmospheric pressure, in spite of the fact that the initial carbon content was higher than that required to maintain equilibrium with the oxygen present. This could be accounted for by the tendency of carbon monoxide to remain in solution at the higher gas pressures. When the total gas pressure was reduced to 0.01 mm. of mercury, however, oxygen contents of the order of 0.001% were obtained after only ten minutes' melting.

#### Metal-Crucible Reactions

The residual carbon contents obtained by Thomas and Moreau were much lower than could be accounted for by a simple reaction between dissolved carbon and oxygen. With ingot 1, a residual carbon content of 0.14% might have been expected if the reaction had proceeded to completion. The final carbon content was, in fact, 0.025%. With ingot 2, an estimated reaction limit of 0.215% of carbon was found to be 0.08%. These anomalies were attributable to metal-crucible reactions which proceeded in parallel with the main reduction process. The final oxygen and carbon contents depended upon the balance arrived at between the following reactions :—



Some results of Moore,<sup>162</sup> summarised in Fig. 7, illustrate the effect of metal-crucible reaction in a characteristic manner. The curves show the variations of oxygen and carbon contents during the vacuum induction melting of iron in a magnesia crucible. A sudden rise in the oxygen content occurs when the carbon content falls below 0.002%. Although the severity of metal-crucible reactions may vary considerably, the curves shown in Fig. 7 illustrate in a qualitative manner the effects which occur whenever metal is heated at low pressure in contact with an oxide refractory.

Hochmann,<sup>124-125</sup> who experimented with carbon-containing chromium irons obtained similar results. His alloys, containing 25-30% of chromium, were vacuum melted in beryllia, magnesia, or alumina crucibles, thereby reducing the carbon contents from 0.030% to 0.002%. In magnesia crucibles the oxygen content could not be reduced below 0.01%. In beryllia or alumina it was practically eliminated. The limiting oxygen content

obtained by Hochmann in a magnesia crucible was ten times that previously reported by Thomas and Moreau. This high value is probably attributable to the great affinity of chromium for oxygen. Hochmann found that the oxidising potential of magnesia *in vacuo* was, in fact, so high that carbon could not be used as an intentional alloying addition when this refractory was employed.

Observations such as these indicate that the final balance between carbon, oxygen and metal will be dictated largely by the crucible material employed. Kubachewski<sup>163</sup> has treated the vacuum melting of pure iron from thermodynamic aspects. Much of the difficulty involved is due to the high stability of dilute

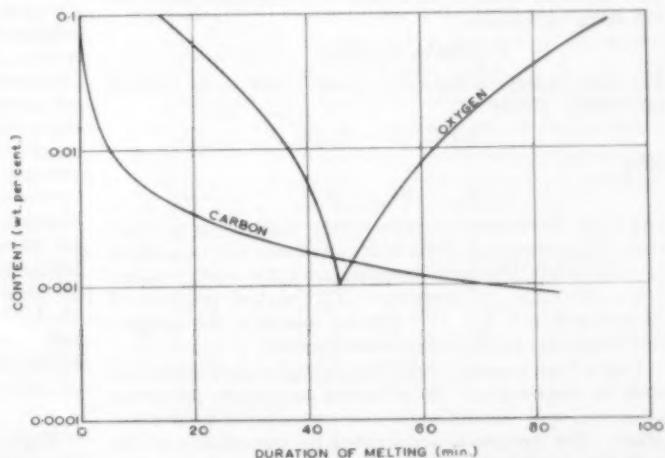


Fig. 7.—Changes of oxygen and carbon contents during the vacuum melting of steel, illustrating the effect of metal-crucible reaction. (J. H. Moore. Courtesy of "Metal Progress".)

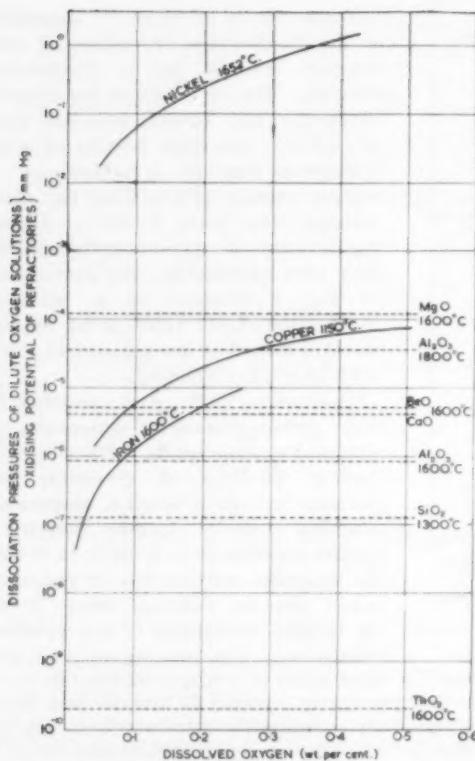


Fig. 8.—Dissociation pressures of dilute solutions of oxygen in molten metals compared with the oxygen potential of typical refractories.

solutions of oxygen in many molten metals. The dissociation pressures of such solutions can be comparable to, and are in many instances much lower than the oxidising potential of the refractory material with which the melt comes into contact.

Thomas and Moreau used magnesia as a refractory. This material tends to dissociate in accordance with the following expression.



The free energy of the reaction at 1,600°C. is 169,500 cal./mole. Hence

$$\log K_p = -19.75$$

Now

$$K_p = P_{\text{Mg}}^2 \times P_{\text{O}_2}$$

and since the vapour pressure of the magnesium is twice that of the oxygen, equilibrium conditions will be arrived at only when the total pressure over the melt reaches  $3.6 \times 10^{-4}$  mm. of mercury. The partial pressure of oxygen will be  $1.2 \times 10^{-4}$  mm. of mercury, the remainder being due to the magnesium vapour.

Under high vacuum conditions magnesium vapour will soon be disposed of. Equilibrium conditions are never achieved, and the refractory is encouraged to disintegrate. The process is accelerated by the affinity of the melt for small traces of oxygen. The partial molar free energy of solution of oxygen in an iron bath already containing 0.2 at. % is 78,000 cal./mole at 1,600°C.

Such a solution is capable of extracting oxygen from any source having a potential higher than  $6 \times 10^{-7}$  mm. of mercury. Since the oxygen potentials of most refractories at 1,600°C. are very much higher than this value, the difficulties sometimes experienced during vacuum melting are readily explicable.

Fig. 8 compares the dissociation pressures of dilute solutions of oxygen in iron, nickel and copper with the oxygen potential at similar temperatures of several refractory materials. It can be concluded that nickel is safely vacuum melted, even in magnesia, with little risk of oxygen pick-up. The ability of iron to extract oxygen from either beryllia, calcium oxide, or alumina, is clearly illustrated. Although the oxidising potential of thoria is very low, expense must preclude the use of this refractory for all except small scale experimental work.

An attempt to obtain quantitative data on metal-crucible reactions was made by Bianchi in 1954. The technique adopted was to follow the rate of deoxidation of baths of iron, held in magnesia crucibles, over the surface of which a continuous current of very pure hydrogen was passed.<sup>164,165</sup> Further studies *in vacuo* gave rather inconsistent results.<sup>165</sup>

An interesting series of trials carried out by Bosworth<sup>167</sup> indicated that the oxidising potential of some refractory crucibles was not a simple function of the stability of the oxide from which they were composed, but was considerably influenced by the minor impurity content. The effect was particularly marked with magnesia.

Charges of pure iron, weighing 200 g. and containing initially 0.003% of carbon and 0.0042% of oxygen, were induction melted under a pressure of approximately 10 microns in commercial crucibles of high purity alumina, zirconia, and magnesia. Some of the crucibles had been pretreated for considerable periods in hydrogen at 1,700°C. Samples taken from the metal bath at short intervals during the melting cycle indicated that the oxygen content achieved a substantially constant value a few minutes after complete fusion. Table II summarises some of the results obtained with treated and untreated crucibles.

An interesting feature of these results is that although the iron picked up a great deal of oxygen from all three refractories the effect was least pronounced with magnesia. The findings contradict, therefore, those of Hochmann, who found alumina to be a far more stable refractory than magnesia. Bosworth concluded that the oxidising potential of the refractories was largely determined by the suboxides and impurity oxides present. Compounds such as  $\text{FeAl}_2\text{O}_4$  could be readily formed by interaction between iron oxide and the alumina crucible material. Such compounds have free energies which may be as low as -10 k. cal./mole at the temperatures under consideration, and could therefore be readily reduced by molten iron. Hydrogen reduction at 1,700°C. would tend to eliminate such unstable compounds. The following reaction was suggested as being typical of those which occur :-



Reduction processes such as these would minimise the quantities of oxygen readily available in the body of the refractory. The general shrinkage and consolidation which occurs during high temperature hydrogen firing

TABLE II.—OXYGEN CONTENTS OF IRON VACUUM MELTED IN OXIDE REFRACRY CRUCIBLES. (BOSWORTH).

|                   | Alumina | Zirconia | Magnesia |
|-------------------|---------|----------|----------|
| Untreated . . . . | 0.0208% | 0.020%   | 0.0156%  |
| Treated . . . .   | 0.0192% | 0.0180%  | 0.0082%  |

may also contribute towards the improved behaviour of the magnesia after this treatment. The author visualises an attack or "etching" of the crucible walls during the melting process. Attainment of oxygen equilibrium could reasonably coincide with the establishment of a constant interface area between charge and crucible.

Although the effect of hydrogen treatment upon the subsequent behaviour of magnesia crucibles is of considerable interest, and possibly of fundamental importance, the improvement in performance, as indicated by the lowered oxygen contents, can only be regarded as marginal. The lowest oxygen value obtained, for example, was 0.0082%, approximately eight times higher than those reported by Thomas and Moreau, and by Moore. It must be emphasised, however, that the iron used by Bosworth had an inherently low carbon content, which would have been insufficient to minimise the rate of oxygen absorption.

The maintenance of a low oxygen content is in fact best assured by reliance upon a fairly high carbon value. Because equilibrium conditions need never be approached, a low oxygen content can be arrived at by casting the ingot after a melting cycle of suitable duration. Although this technique is practised on an industrial scale with considerable success the basic mechanisms involved are still not fully understood. The authors of a recent paper on the carbon-oxygen relationships in molten iron made significant progress towards a solution of some of these problems.

Parlee, Seagle and Schuhmann<sup>168</sup> concluded that a true understanding of the relationships between carbon, oxygen, and molten iron could not be arrived at without reference to metal-crucible reactions. Under limiting conditions the rate of the metal-crucible reaction was found to be similar to that of the reaction taking place at the gas-metal interface. A sensitive index of the extent of the reaction under vacuum conditions was afforded by the amount of dissolved aluminium taken up by the melt from the alumina crucible. The effect could be demonstrated visually in a very simple manner by admitting carbon monoxide above the surface of a molten charge previously degassed in *vacuo*. A thick crust was almost instantly formed on the surface of the molten metal. This crust consisted of finely divided alumina, formed by the sudden reaction between dissolved aluminium and the carbon monoxide atmosphere.

The authors found that the results of their determinations were explicable on the assumption that limiting conditions occurred at both gas-metal and metal-crucible interfaces. Dissolved carbon and oxygen had to come to a surface to react. The existence of relatively "stagnant" films at the gas-metal, and metal-crucible interfaces was postulated, the rate of carbon-oxygen reaction being controlled by the rate of oxygen diffusion through these boundary layers.

No determinations of the oxygen content of the iron were made by these investigators, who followed the rate of progress of the reaction between the liquid metal, carbon monoxide, and alumina crucible by means of the

changes of gas volume at constant pressure. The effect of gas pressure on the rate of evolution of carbon monoxide was found to agree with theoretical predictions as to the effect of gas pressure on the following reaction:



The working and mechanical properties of certain high duty nickel based alloys are profoundly affected by traces of boron and zirconium introduced from the crucible during vacuum melting. In the experiments reported,<sup>194</sup> boron contamination up to 0.008% resulted from melting in slightly impure magnesia crucibles. The zirconia crucibles employed introduced up to 0.19% of zirconium. No contamination resulted from melting in the alumina crucibles employed, which were fairly pure. Alloys melted in these pure oxide crucibles had mechanical properties inferior to those which contained traces of zirconium or boron. Excessive quantities of these two elements in combination, however, impaired hot workability and led to reduced mechanical properties.

(to be continued)

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## A New High Temperature Steel

SAMUEL FOX & CO., LTD., a subsidiary of The United Steel Cos., Ltd., has started commercial production of a new alloy steel designed for service temperatures up to 675° C. Known as Esshete 1250, this austenitic creep-resisting steel is the outcome of five years of research and testing, with the result that unusually comprehensive data is available on its properties and performance.

Esshete 1250 is a 15% chromium, 10% nickel, 6% manganese steel, the composition including smaller percentages of silicon, molybdenum, vanadium, niobium and boron. It is claimed that it combines a high level of rupture strength with adequate ductility, good weldability, structural stability and oxidation resistance at elevated temperatures for long periods. It also has good manipulation properties. The steel is thus admirably suited for service in power stations of advanced design.

Esshete 1250 has proved satisfactory in the production of bars, tubes, pipes and large forgings, and sheet trials are in progress. Large bars, pipes and plate can be readily fabricated in either the hot or cold condition, cold forming being understood to include temperatures not exceeding 650° C. and hot forming not less than 900° C. Whichever method of fabrication is adopted, the material requires a re-solution treatment within the range 1,050–1,150° C.

In extensive weldability trials, employing various experimental and commercial electrodes, Esshete 1250 gave excellent results.

The structural stability of the steel compares most favourably with other more conventional austenitic steels. Its oxidation resistance has been assessed by a combination of laboratory tests and exposure in an experimental element in a power station for a period of approximately two years. In this case, too, Esshete 1250 has shown characteristics similar to those of other austenitic steels of the same chromium content.

The normal mechanical properties of the steel at room and elevated temperatures are shown in Table I. Particular attention has been devoted to the stress-to-rupture properties, the test programme having covered a period of 25,000 hours to date. The results of this extensive testing are summarised in Table II. Creep testing up to 10,000 hours has been carried out to determine the stress to give 0.1% and 0.2% strain, and the results of these tests are summarised in Table III.

Like other austenitic steels in the solution-treated condition, Esshete 1250 has low proof stress values. These can be improved by warm working, and this pro-

TABLE I.—TYPICAL MECHANICAL PROPERTIES IN THE SOLUTION TREATED CONDITION

| Test Temperature (° C.) | Tensile Strength (tons/sq. in.) | Proof Stress (tons/sq. in.) |        | Limit of Proportionality (0.1%) | Elongation (4 $\sqrt{A}$ %) | Reduction of Area (%) | Modulus of Elasticity (tons/sq. in.) |
|-------------------------|---------------------------------|-----------------------------|--------|---------------------------------|-----------------------------|-----------------------|--------------------------------------|
|                         |                                 | (0.1%)                      | (0.2%) |                                 |                             |                       |                                      |
| 29 ..                   | 39.0                            | 13.8                        | 15.0   | 7.0                             | 67.0                        | 89.6                  | 13,400                               |
| 100 ..                  | 34.3                            | 10.6                        | 11.6   | 5.2                             | 64.5                        | 72.0                  | 13,000                               |
| 200 ..                  | 31.8                            | 9.9                         | 10.1   | 4.2                             | 55.0                        | 70.3                  | 12,500                               |
| 300 ..                  | 31.4                            | 8.5                         | 9.4    | 2.6                             | 45.0                        | 84.0                  | 11,900                               |
| 400 ..                  | 31.5                            | 8.6                         | 9.1    | 4.4                             | 47.7                        | 80.8                  | 11,500                               |
| 500 ..                  | 30.3                            | 8.2                         | 8.8    | 4.8                             | 40.5                        | 60.8                  | 11,700                               |
| 600 ..                  | 28.5                            | 8.2                         | 8.6    | 4.8                             | 47.4                        | 64.0                  | 10,200                               |
| 700 ..                  | 23.0                            | 8.1                         | 8.5    | 3.6                             | 40.0                        | 51.2                  | 9,650                                |

TABLE II.—STRESS-TO-RUPTURE TEST RESULTS

| Time (hours) | Stress to Rupture (tons/sq. in.) at |         |         |         |         |
|--------------|-------------------------------------|---------|---------|---------|---------|
|              | 600° C.                             | 625° C. | 650° C. | 675° C. | 700° C. |
| 100 ..       | 24.0                                | 20.5    | 18.0    | 16.3    | 15.5    |
| 300 ..       | 22.0                                | 18.5    | 10.5    | 15.1    | 14.6    |
| 1,000 ..     | 20.0                                | 17.0    | 13.0    | 13.5    | 13.0    |
| 2,000 ..     | 18.5                                | 16.0    | 14.0    | 12.0    | 10.0    |
| 10,000 ..    | 16.8                                | 14.5    | 12.5    | 10.4    | 7.5     |
| 30,000 ..    | (15.0)                              | (13.5)  | (11.4)  | (8.9)   | (5.8)   |
| 100,000 ..   | (13.9)                              | (12.5)  | (10.8)  | (8.2)   | (4.5)   |

TABLE III.—CREEP TEST RESULTS

| Temperature (° C.) | Stress for 0.1% Creep Strain (tons/sq. in.) after |           |            | Stress for 0.2% Creep Strain (tons/sq. in.) after |           |            |
|--------------------|---|-----------|------------|---|-----------|------------|
|                    | 1,000 hr.   | 3,000 hr. | 10,000 hr. | 1,000 hr.   | 3,000 hr. | 10,000 hr. |
| 650 ..             | 7.60  | 6.00      | 5.15       | 7.80  | 7.25      | 6.65       |
| 675 ..             | 6.55  | 5.45      | 4.70       | 7.30  | 6.50      | 5.60       |
| 700 ..             | 4.90  | 3.60      | 1.80       | 5.60  | 5.30      | 3.10       |

cess is found, in fact, to push up the 0.1% proof stress to 32 tons/sq. in. at room temperature and 23 tons/sq. in. at 625° C., while still holding elongations on 4 $\sqrt{A}$  of the order of 48% and 25%, respectively. The hardness of the steel is not altered at 625° C., but at room temperature is the equivalent of 43 tons/sq. in. ultimate tensile strength, which is only slightly more than in the normally rolled condition. Warm working also improves the creep properties, so that the steel is well suited in this condition for use as a high temperature bolting material.

### Savings from Carbide Injection

EXTENSIVE tests have shown that carbide injection equipment supplied by British Oxygen will enable the Aston works of Spartan Steels & Alloys, Ltd., to make fuller use of scrap metal in producing stainless steel. With the carbide injection process the sulphur content of the raw material can be reduced to the required level without having to use relatively expensive pig iron. Equipment consists of a carbide dispenser and a lance. Carbide is blown through the lance and into the melt by compressed nitrogen.

Sulphur content in a 3-ton melt can be reduced from 0.078% to 0.024% in twenty minutes, using 200 lb. of carbide and 200 cu. ft. of nitrogen. It is claimed that other fluxing agents would take at least two hours at several times the cost. At present, Spartan Steels are using carbide injection on one 3-ton furnace. Their second 3-ton furnace will soon be employing the technique and ultimately both will be doing six melts a day, using carbide injection.

### Gas Cleaning at Arc Furnace Plant

WHEN the Steel, Peech & Tozer branch of the United Steel Cos., Ltd., announced their £10 million scheme for the replacement of all existing open hearth steel melting furnaces by six 110-ton oxygen-lanced electric arc furnaces, it was stated that special attention would be given to the selection of fume extraction equipment. An order has now been placed with the gas cleaning division of W. C. Holmes & Co., Ltd., to supply gas cleaning plant for each of the first two furnaces to be installed at Templeborough Works. Each gas cleaning installation consists of a direct fume extraction system, an electrical precipitator and a common water treatment plant.

# The Ferritisation of S.G. Cast Iron by the Controlled-Cooling Process

By G. J. Cox, A.C.T.(B'ham), A.I.M.\*

*The development in S.G. iron of the ferritic matrix necessary where increased ductility and shock resistance are required is achieved commercially by (a) the three-stage process, (b) the two-stage process, and (c) controlled-cooling. The first two methods involve holding at 700° C. for a prolonged period, and the controlled-cooling technique would appear to offer advantages on the grounds of cost if the permissible cooling rate were high enough. This speed depends on the composition of the iron, the amount of pearlite permissible for maximum ductility, and the as-cast section size. The author presents here the results of an investigation undertaken to study the first two of these factors.*

## Introduction

**I**N the as-cast condition S.G. iron usually has a matrix structure consisting mainly of pearlite, and when increased ductility and shock-resistance are required it is necessary to develop a ferritic matrix by some form of heat treatment.

The various treatments which will produce a micro-structure consisting of graphite in a matrix of ferrite consist essentially of two parts: (1) austenitisation, and (2) ferritisation. It was formerly believed that the main purpose of austenitisation was to eliminate any eutectic carbides which might have formed during solidification, and that if it was unlikely that carbide would be present in the casting, ferritisation could be achieved by merely holding for a suitable period at a sub-critical temperature, e.g. 700° C. However, as shown by Gilbert,<sup>1</sup> if maximum impact values are to be obtained, prior austenitisation is essential. The various annealing heat treatments which are commercially applied to S.G. iron may be described as follows:

(1) *The Three-Stage Process*—This treatment consists in austenitising the castings for an appropriate period, e.g. 2–8 hours, furnace-cooling at the most convenient rate to about 700° C., and holding for 4–16 hours at that temperature. The castings are then sometimes furnace-cooled to 300°–400° C. and finally air-cooled, but more generally they are simply air-cooled from about 650° C.

Some ferritisation of the austenite can take place during cooling over the range 800° C. to about 750° C., and ferritisation of pearlite is effected by holding at 700° C. Clearly, the amount of pearlite to be ferritised at 700° C., and hence the holding time needed at this temperature, will depend on the rate of cooling from 800° C. This process may therefore be described as a three-stage treatment.

(2) *The Two-Stage Process*—This treatment comprises austenitising and then air-cooling to room temperature. The castings are then re-charged into a furnace held at about 700° C. for ferritisation. When this process is used the ferrite forms primarily from pearlite.

(3) *The Controlled-Cooling Process*—This consists in cooling at a controlled rate from the austenitising temperature down to 650° C. The castings are then removed and air-cooled. In this case the ferrite will form primarily from austenite as the iron passes through the eutectoid range.

To date, the controlled-cooling method is probably least often used and its value has not yet been fully assessed.

## Relative Merits of the Ferritising Processes for S.G. Iron

All the processes summarised above have received study by previous investigators and it is known that practically complete elimination of pearlite, accompanied by satisfactory mechanical properties, can be achieved by any one of the three methods. Their relative merits therefore depend upon comparative cost, which is, of course, mainly dependent upon the time needed for treatment. However, it has been clearly shown that in the treatment of S.G. irons ferritisation of austenite is more rapid than that of pearlite,<sup>2</sup> and it would seem therefore that the controlled-cooling method is to be preferred.

Despite these considerations, Millis<sup>3</sup> and Savage<sup>4</sup> suggest that when using the controlled-cooling process the maximum cooling speed permissible through the eutectoid range (790°–650° C.) is 19° C./hour, and Perry and Rehder<sup>5</sup> conclude that the maximum allowable speed is 45° C./hour. It is clear that limits of this order preclude the use of very rapid ferritisation processes. On the other hand, Hodgson and Fairhurst,<sup>6</sup> and more recently Eckel<sup>7</sup>, have found that cooling speeds considerably in excess of even 45° C./hour could result in almost complete elimination of pearlite.

The maximum cooling speed that can be used will be dependent primarily upon (1) the composition of the iron; (2) the amount of pearlite which can be tolerated in the microstructure of the iron, concomitant with obtaining maximum ductility; and (3) the as-cast section size. The present investigation was undertaken to study the first two of these factors.

## Materials Used

The compositions of the irons used are given in Table I. These irons are typical of those likely to be encountered

\* The International Nickel Co. (Mond), Ltd., Development and Research Department Laboratory, Birmingham.

TABLE I.—COMPOSITION, MICROSTRUCTURE AND HARDNESS OF THE IRONS "AS-CAST"

| Iron No. | Composition |        |        |        |       |        |        | Microstructure |                 | Brinell Hardness                        |           |
|----------|-------------|--------|--------|--------|-------|--------|--------|----------------|-----------------|---|-----------|
|          | C (%)       | Si (%) | Mn (%) | Si (%) | P (%) | Ni (%) | Cr (%) | Mg (%)         | Graphite        |   |           |
| 1        | 3.20        | 2.00   | 0.20   | 0.01   | 0.01  | <0.10  | —      | 0.056          | 90% S.G. + C.G. | 45-5% ferrite, remainder pearlite       | 188       |
| 2        | 3.28        | 2.00   | 0.54   | 0.01   | 0.01  | <0.10  | —      | 0.051          | 90% S.G. + C.G. | 21-7% ferrite, remainder pearlite       | 225       |
| 3        | 3.22        | 1.80   | <0.10  | 0.01   | 0.01  | 0.58   | —      | 0.058          | 90% S.G. + C.G. | 37-0% ferrite, remainder pearlite       | 199       |
| 4        | 3.20        | 2.00   | 0.20   | 0.01   | 0.01  | 1.70   | —      | 0.055          | 90% S.G. + C.G. | Estimate 3% ferrite, remainder pearlite | About 280 |
| 5        | 3.31        | 2.00   | 0.34   | 0.01   | 0.01  | <0.10  | 0.25   | 0.063          | 90% S.G. + C.G. | 4-3% ferrite, remainder pearlite        | 240       |
| 6        | 3.20        | 2.00   | 0.35   | 0.01   | 0.01  | 0.85   | —      | 0.060          | 90% S.G. + C.G. | Estimate 3% ferrite, remainder pearlite | About 250 |

S.G. Spheroidal Graphite.

C.G. Compacted Graphite.

\* Chemical determination: the other elements were determined spectrographically.

† Nominal analysis of base iron.

‡ The samples produced to this composition were too numerous to make in one melt: the analysis shown is therefore that typical of several casts.

industrially, and all except No. 3 were made from Warner S.P.H. pig iron. For iron No. 3, Bremanger oxygen-refined pig iron was used.

The nickel-free irons were made by plunging the melt with magnesium turnings at 1,400°C., the nickel-containing irons by tapping the metal at 1,420°C. onto a 15% Mg-Ni alloy. In both cases inoculation with ferro-silicon (80% Si) followed, and finally the metal was poured into green-sand moulds, to produce 1 in. thick keel-block castings. As-cast microstructures are given in Table I. (In this and all subsequent cases the amount of pearlite present was determined by linear analysis, using a phase-counting device.)

### Experimental Procedure and Results

#### Effect of Cooling Rate on Microstructure and Hardness

This part of the work was confined to Irons Nos. 1-5. Samples  $\frac{1}{2}$  in. square were heated for 2 hours at 900°C., in a tubular resistance furnace, and were then cooled linearly from 900° to 650°C. at 5, 15, 40, 70, 100, 130, 160 or 200°C./hour. Specimens so treated were subjected to microscopic examination and hardness testing. The effect of cooling rate on pearlite content is shown in Fig. 1 and its influence on hardness in Fig. 2.

As would be expected, the degree of ferritisation obtained is dependent upon the composition of the iron. Thus nickel, manganese and chromium are seen to retard

the ferritisation, the relative effects of these elements being similar to those found in previous work on the three-stage process.<sup>8</sup> In normal practice probably only Irons Nos. 1-4 would be considered suitable for annealing.

A check was made on ferrite grain-size of the samples cooled at the respective speeds, but no significant differences were found.

As stated above, in these tests linear cooling rates were used from 900°C., but it has been implied by other investigators that when using the controlled-cooling process the cooling rate between 900° and 790°C. is unimportant, since the critical range for ferritisation lies between 790° and 650°C.

Tests were first made to check the effect of rapid cooling from 900°C. to the upper temperature of the ferritising range. The irons were austenitised for 2 hours at 900°C., and then rapidly cooled to (a) 800°C. or (b) 750°C. All specimens were then cooled at 70°C./hour to 600°C. The microstructures so produced are detailed in Table II. Comparison with the results of the linear cooling tests shows that very rapid cooling to 800°C. has no detrimental effect, but that the same rate of cooling to 750°C. leads to somewhat less satisfactory results.

Thus, for practical purposes the upper critical temperature for irons of the compositions studied may be taken to be 800°C., but its exact position will depend upon (a) the composition of the iron and (b) the speed of

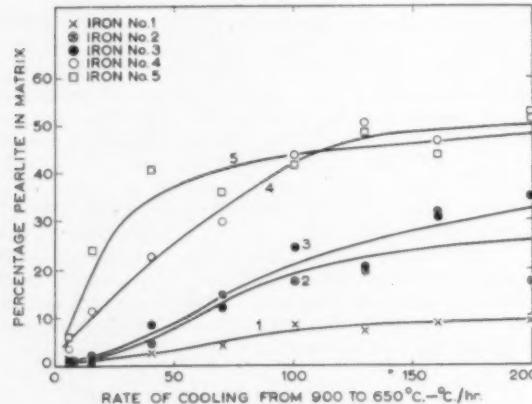


Fig. 1.—Effect of controlled-cooling rate on the amount of pearlite formed.

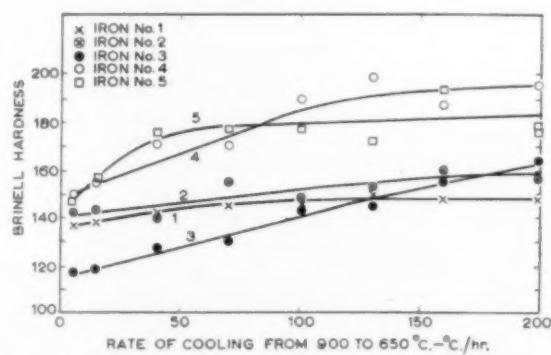


Fig. 2.—Effect of controlled-cooling rate on the Brinell hardness.

TABLE II.—EFFECT OF INITIAL RAPID COOLING FROM 900°C. TO INTERMEDIATE TEMPERATURES ON THE MICROSTRUCTURES OF IRONS SUBSEQUENTLY COOLED AT 70°C./HOUR TO 650°C.

| Treatment   | Specimen Iron No.     | Composition of Matrix (pearlite % : remainder ferrite) |
|---|-----------------------|--|
| Specimens held at 900°C. for 2 hours; furnace-cooled at approximately 1,000°C./hour to 750°C., and then cooled at 70°C./hour to 650°C., followed by air-cooling | 1<br>2<br>3<br>4<br>5 | 6.0<br>19.8<br>26.9<br>38.3<br>61.0                    |
| Specimens held at 900°C. for 2 hours; furnace-cooled at approximately 1,000°C./hour to 800°C., and then cooled at 70°C./hour to 650°C., followed by air-cooling | 1<br>2<br>3<br>4<br>5 | 4.3<br>14.0<br>11.5<br>24.5<br>37.4                    |

cooling. The effect of these factors has been indicated by Rehder.<sup>9</sup>

Since it was considered likely that the recommended finishing temperature of 650°C. was unnecessarily low, tests were made to check this point and to provide additional information on the microstructural changes occurring during the cooling process. Specimens of Irons Nos. 1, 2 and 3 were cooled from 900°C. at 50°C./hour and were water-quenched from selected temperatures. The results of microscopic examination and hardness testing of these samples are given in Figs. 3, 4 and 5. Again it is indicated that the upper critical point may be taken as approximately 800°C. and also that, contrary to previous recommendations, cooling need not be continued below 700°C. before extraction of the casting and air-cooling. In commercial practice, however, some margin is necessary and castings may well be left to cool in industrial furnaces to 650°C. before withdrawal.

The results already obtained indicate the effect of manganese on the response of nickel-free irons (i.e. Irons Nos. 1 and 2, Fig. 1). Further tests were made to study the effect of continuous variation of the major elements on the response to ferritisation of the nickel-containing S.G. irons. For these tests a standard cooling speed considered appropriate for a 2.0% Si, 0.30% Mn, 0.85% Ni iron was used.

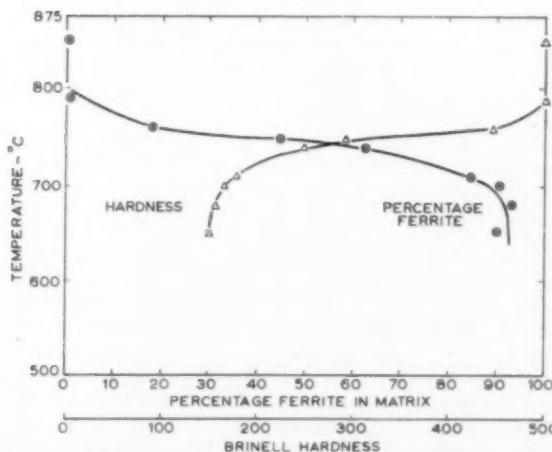


Fig. 4.—Microstructure and hardness of specimens of Iron No. 2 quenched during controlled-cooling at 50°C./hr.

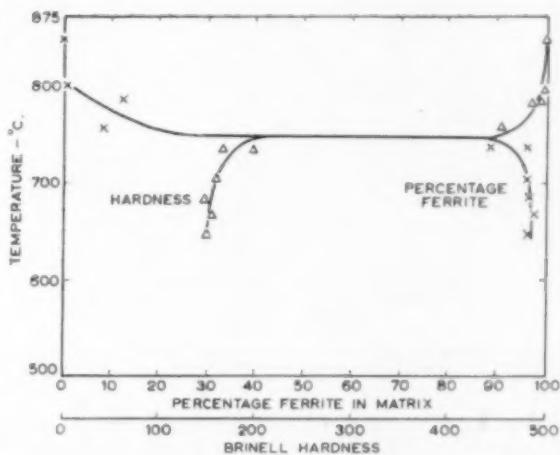


Fig. 3.—Microstructure and hardness of specimens of Iron No. 1 quenched during controlled-cooling at 50°C./hr.

#### Effect of Silicon, Manganese, Nickel and Magnesium on the Ferritisation of Irons Continuously Cooled at 50°C./hour.

The analyses of the irons used are shown in Table III, together with the results obtained after treatment. Figs. 6, 7 and 8 show, respectively, the effects of silicon, manganese and nickel. The effect of silicon was determined on three types of base iron. All the specimens were austenitised for 2 hours at 900°C. in an electric furnace, and cooled at 50°C./hour from 800° to 680°C., from which temperature they were air-cooled.

As shown by Fig. 6, silicon has a marked effect on the amount of ferrite so obtained, and if this heat treatment is applied to highly alloyed irons, such as those corresponding to curves B and C in the figure, the silicon content has a particularly critical effect. Thus, Eckel<sup>6</sup> studied the ferritisation of an iron similar to Iron No. 4 (Table 1), and obtained satisfactory ferritisation at

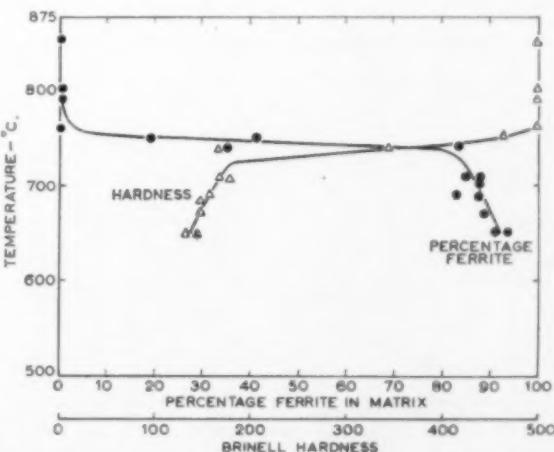


Fig. 5.—Microstructure and hardness of specimens of Iron No. 3 quenched during controlled-cooling at 50°C./hr.

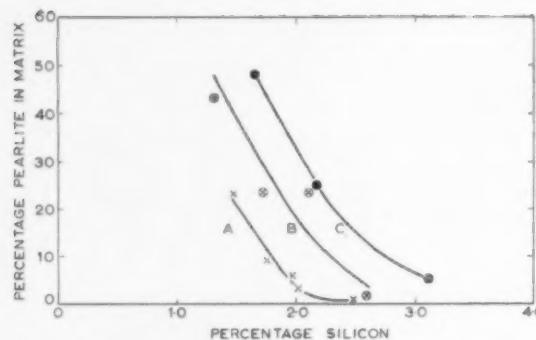


Fig. 6.—Effect of silicon content on the amount of pearlite formed on controlled-cooling at 50° C./hr. (see Table III).

cooling rates much higher than those used in the present investigation. The silicon content of his specimens was, however, 0.5% higher, i.e., 2.4-2.6%, and this is the probable explanation for the discrepancy. It must be remembered, however, that whilst a high silicon content in S.G. iron assists ferritisation, this element adversely affects the impact properties and, for this reason, should not normally exceed 2.5%.

Much of the S.G. iron currently produced contains silicon 2.0-2.2%, nickel 0.8-1.0%, and manganese 0.30%, and Fig. 7 shows that, as indicated by the results of the initial experiments, a cooling speed of 50° C./hour would probably effect satisfactory ferritisation of such an iron. It is also clear, however, that if the manganese content were raised, the use of slightly slower cooling speeds would become necessary.

Nickel has a less marked effect than manganese, but Fig. 8 indicates that, at the selected cooling speed, about 1.00% nickel is the maximum which can be present without formation of increased amounts of pearlite. It has already been demonstrated that in the absence of nickel very fast cooling rates (up to 80° C./hour) may be used (Iron No. 1, Fig. 1), whilst with a high nickel content (1.5% or above) cooling speeds of less than 15° C./hour must be employed to obtain satisfactory results (Iron No. 4, Fig. 1).

Magnesium has no effect on the response to ferritisation (Table III).

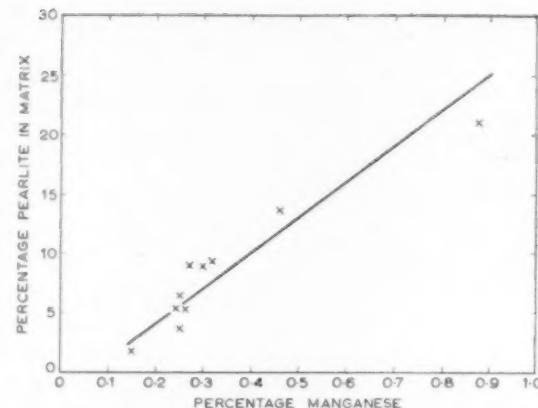


Fig. 7.—Effect of manganese content on the amount of pearlite formed on controlled-cooling at 50° C./hr. (see Table III).

*Comparison of the Mechanical Properties of S.G. Iron Ferritised by the Three-Stage Process and by Controlled-Cooling, and the Effect of Residual Pearlite Content.*

The object of these final tests was twofold:—

- (1) To make simple comparisons between the mechanical properties of irons ferritised by the three-stage process and by a controlled-cooling process.
- (2) To determine the critical pearlite content in S.G. iron above which the ductility might be expected to deteriorate.

A large number of keel-block castings were produced from an iron of composition corresponding to No. 6 (Table I): one group was ferritised by three-stage processes, the other by controlled-cooling processes. The tensile and impact properties of the irons so treated were determined and sections were cut from each specimen for microscopical examination and hardness testing. (The tensile specimens had a diameter of 0.564 in. and a gauge length of 2 in.: the impact tests were made on 0.450 in. diameter notched Izod specimens.)

Details of the treatments and the results are shown in Tables IV and V. The values given indicate no significant difference between the mechanical properties obtained by the two treatments, although the continuously-cooled samples generally contained slightly more pearlite than those which had been annealed by the conventional procedure.

It is, however, clear, that when a certain critical quantity of pearlite is present in the microstructure of S.G. iron, ductility is impaired. Obviously, in the last test this level had not been reached, although the cooling speed used was considered a marginal one for this particular composition, and was indeed the fastest that the furnace was capable of giving. It is obviously important that this critical level should be established, since it will determine the maximum permissible cooling rate.

In order to determine this critical value, keel blocks of Iron No. 4 were austenitised and then cooled to 690° C. at 50° C./hour, at which stage some of the specimens were extracted and air-cooled, whilst others were held for various lengths of time at 690° C. before air-cooling.

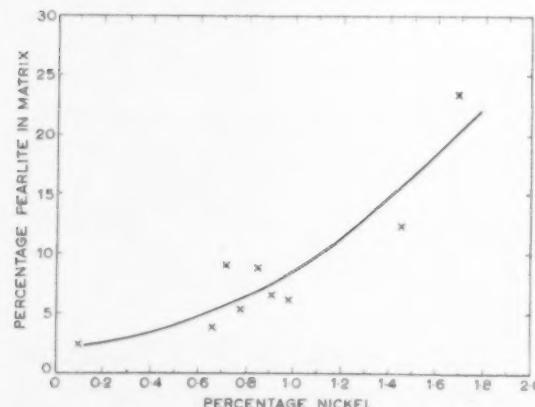


Fig. 8.—Effect of nickel content on the amount of pearlite formed on controlled-cooling at 50° C./hr. (see Table III).

TABLE III.—EFFECT OF SILICON, MANGANESE, NICKEL AND MAGNESIUM ON THE MICROSTRUCTURE OF IRONS AUSTENITISED FOR 2 HOURS AT 900° C. AND THEN CONTINUOUSLY COOLED AT 50° C./HOUR TO 680° C.

| Description                                     | Composition  |  |  |  |  | Percentage Pearlite in Matrix (remainder ferrite) |
|---|--|--|--|--|--|---|
|   | C (%)  | Si (%)   | Mn (%)   | Ni (%)   | Mg (%)   |   |
| <b>SILICON SERIES</b>                           |  |  |  |  |  |   |
| (A) Mn 0.20-0.25%<br>Ni 0.63-0.98%              | 1.47<br>1.75<br>1.95<br>2.00<br>2.47   | 0.20<br>0.29<br>0.24<br>0.25<br>0.23   | 0.68<br>0.69<br>0.98<br>0.66<br>0.79   | 0.083<br>0.070<br>0.105<br>0.048<br>0.084  | 23.2<br>9.4<br>5.6<br>3.9<br>0.35                                    |   |
| 3.20-3.40%                                      |  |  |  |  |  |   |
| (B) Mn 0.27-0.32%<br>Ni 1.65-1.75%              | 1.30<br>1.72<br>2.10<br>2.60   | 0.27<br>0.29<br>0.30<br>0.32   | 1.65<br>1.70<br>1.70<br>1.75   | 0.061<br>0.082<br>0.055<br>0.105   | 45.3<br>23.4<br>23.5<br>1.9  |   |
| 3.20-3.40%                                      |  |  |  |  |  |   |
| (C) Mn 0.83-1.06%<br>Ni 0.61-0.71%              | 3.46<br>3.53<br>3.58   | 0.65<br>0.77<br>1.06   | 0.83<br>0.80<br>0.71   | 0.060<br>0.067<br>0.063  | 48.5<br>24.9<br>5.2  |   |
| 3.20-3.40%                                      |  |  |  |  |  |   |
| <b>MANGANESE SERIES</b>                         |  |  |  |  |  |   |
| Si 1.90-2.17%<br>Ni 0.61-0.98%                  | 2.00<br>1.95<br>2.00<br>2.10<br>1.90<br>1.90<br>1.90<br>1.95<br>1.90<br>2.17 | 0.15<br>0.24<br>0.25<br>0.25<br>0.26<br>0.27<br>0.29<br>0.32<br>0.46<br>0.88 | 0.79<br>0.98<br>0.66<br>0.91<br>0.78<br>0.72<br>0.85<br>0.85<br>0.68<br>0.61 | 0.055<br>0.105<br>0.048<br>0.062<br>0.051<br>0.060<br>0.056<br>0.145<br>0.076<br>0.067 | 2.0<br>5.6<br>3.9<br>6.5<br>5.4<br>9.2<br>9.0<br>9.5<br>13.8<br>20.8 |   |
| 3.20-3.40%                                      |  |  |  |  |  |   |
| <b>NICKEL SERIES</b>                            |  |  |  |  |  |   |
| Si 1.90-2.10%<br>Mn 0.24-0.30%                  | 2.00<br>2.00<br>1.90<br>1.90<br>2.10<br>1.95<br>1.95<br>2.10<br>2.10<br>2.10 | 0.30<br>0.25<br>0.27<br>0.28<br>0.25<br>0.24<br>0.24<br>0.30<br>0.30<br>0.30 | 0.10<br>0.66<br>0.72<br>0.78<br>0.91<br>0.85<br>0.85<br>1.45<br>0.98<br>0.61 | 0.056<br>0.048<br>0.060<br>0.051<br>0.062<br>0.056<br>0.076<br>0.071<br>0.071<br>0.067 | 2.4<br>3.9<br>9.2<br>5.4<br>6.5<br>9.0<br>12.6<br>12.6<br>23.5       |   |
| 3.20-3.40%                                      |  |  |  |  |  |   |
| <b>MAGNESIUM SERIES</b>                         |  |  |  |  |  |   |
| Si 1.75-2.10%<br>Mn 0.20-0.32%<br>Ni 0.66-0.98% | 2.00<br>1.90<br>1.90<br>1.90<br>2.10<br>1.75<br>1.95<br>1.95<br>1.95<br>1.95 | 0.25<br>0.26<br>0.29<br>0.27<br>0.25<br>0.20<br>0.21<br>0.24<br>0.32         | 0.66<br>0.78<br>0.85<br>0.72<br>0.91<br>0.69<br>0.98<br>0.98<br>0.85         | 0.048<br>0.051<br>0.056<br>0.060<br>0.062<br>0.070<br>0.105<br>0.105<br>0.145          | 3.9<br>5.4<br>9.0<br>9.2<br>6.5<br>9.4<br>3.2<br>5.6<br>9.5          |   |
| 3.20-3.40%                                      |  |  |  |  |  |   |

\* Nominal content.

(The tests were made on Iron No. 4 because the earlier experiments had shown that this composition would give an appreciable quantity of pearlite after continuous cooling at 50° C./hour.) A large number of keel blocks of Iron No. 6 also were similarly treated.

TABLE IV.—TENSILE PROPERTIES, HARDNESS AND MICROSTRUCTURE OF IRON NO. 6 AFTER TREATMENT BY A THREE-STAGE PROCESS AND BY CONTROLLED-COOLING

| Designation                        | Description of Process  | Specimen No. | Mechanical Properties                  |                                  |   |                         |                                 | Percentage Pearlite in Matrix (remainder ferrite) |
|------------------------------------|---|--------------|--|----------------------------------|---|-------------------------|---------------------------------|---|
|                                    |   |              | Ultimate Tensile Stress (tons/sq. in.) | 0.2% Proof Stress (tons/sq. in.) | Limit of Proportionality (tons/sq. in.) | Elongation on 2 in. (%) | Brinell Hardness (120 kg. load) |   |
| Conventional Three-Stage Treatment | Specimens 1-4 were held for 4 hr. at 900° C., furnace-cooled to 680° C. at 50° C./hr.; held 16 hr. at 680° C. and air-cooled. | 1            | 20.1                                   | 23.6                             | 16.1                                    | 29.0                    | 161                             | 0.4   |
|                                    | Specimens 5 and 6 were held for 16 hr. at 900° C., and for 48 hr. at 680° C. before air-cooling                               | 2            | 20.1                                   | 23.2                             | 15.6                                    | 29.0                    | 160                             | 0.9   |
|                                    |   | 3            | 31.2                                   | 23.5                             | 15.2                                    | 24.0                    | 168                             | 2.6   |
|                                    |   | 4            | 30.8                                   | 25.2                             | 16.1                                    | 27.0                    | 156                             | 3.8   |
|                                    |   | 5            | 28.0                                   | 20.6                             | 15.4                                    | 28.0                    | 145                             | Trace   |
|                                    |   | 6            | 31.9                                   | 23.5                             | 16.2                                    | 24.0                    | 162                             | 4.2   |
| Controlled-Cooling Treatment       | The specimens were held for 4 hr. at 900° C., furnace-cooled to 650° C. at 50° C./hr. and then air-cooled.                    | 1            | 20.3                                   | 22.3                             | 16.0                                    | 27.0                    | 163                             | 0.6   |
|                                    | Specimen No. 10 was cooled at 100° C./hr. from 900° C. to 800° C.   | 2            | 20.3                                   | 22.0                             | 16.0                                    | 27.0                    | 162                             | 0.1   |
|                                    |   | 3            | 20.2                                   | 21.8                             | 14.0                                    | 26.0                    | 163                             | 5.2   |
|                                    |   | 4            | 30.1                                   | 22.7                             | 17.0                                    | 28.0                    | 164                             | 3.2   |
|                                    |   | 5            | 29.8                                   | 22.4                             | 17.0                                    | 26.0                    | 167                             | 4.2   |
|                                    |   | 6            | 30.1                                   | 22.8                             | 16.6                                    | 27.0                    | 162                             | 7.4   |
|                                    |   | 7            | 30.3                                   | 21.6                             | 15.0                                    | 28.0                    | 161                             | 4.6   |
|                                    |   | 8            | 29.9                                   | 22.9                             | 14.0                                    | 25.0                    | 158                             | 2.0   |
|                                    |   | 9            | 20.4                                   | 22.6                             | 14.1                                    | 27.0                    | 158                             | 2.1   |
|                                    |   | 10           | 29.0                                   | 20.5                             | 14.9                                    | 28.0                    | 146                             | 5.9   |

TABLE V.—IMPACT PROPERTIES, HARDNESS AND MICROSTRUCTURE OF IRON NO. 6 AFTER TREATMENT BY A THREE-STAGE PROCESS AND BY CONTROLLED-COOLING

| Designation                        | Description of Process   | Specimen No. | Impact Values (ft. lb.) | Brinell Hardness (120-kg. load) | Percentage Pearlite in Matrix (remainder ferrite) |
|------------------------------------|--|--------------|-------------------------|---------------------------------|---|
| Conventional Three-Stage Treatment | Specimens held for 4 hr. at 900° C., furnace-cooled to 680° C. at 50° C./hr.; held 16 hr. at 680° C. and air-cooled. | 1            | 14, 14, 15              | 155                             | 1.0   |
|                                    |  | 2            | 14, 14, 14              | 152                             | 1.4   |
|                                    |  | 3            | 14, 14, 13              | 147                             | 0.5   |
|                                    |  | 4            | 14, 15, 14              | 146                             | 0.5   |
| Controlled-Cooling Treatment       | Specimens held for 4 hr. at 900° C., furnace-cooled to 650° C. at 50° C./hr., and then air-cooled.                   | 1            | 14, 15, 15              | 152                             | 2.8   |
|                                    |  | 2            | 14, 14, 14              | 153                             | 1.6   |
|                                    |  | 3            | 14, 13, 13              | 156                             | 4.2   |
|                                    |  | 4            | 13, 14, 13              | 144                             | 3.6   |

These treatments produced specimens having various residual pearlite contents and again the tensile and impact properties were determined and the amount of pearlite present in each specimen was found by linear analysis. As a means of conveniently summarising the results, two ductility criteria were used: tensile elongation and impact values as percentages of the maximum values obtained for each of the irons, and these percentage values were plotted against pearlite content. The results are shown in Figs. 9 and 10. In each figure the horizontal line represents the average percentage value obtained from irons containing less than 3.0% pearlite.

The figures show that in some instances abnormally high pearlite contents were found in both these irons. This is due to variations in composition, particularly in silicon and nickel contents, but these differences were relatively slight (0.25-0.30%) and would not, *per se*, have any significant effect on mechanical properties. The results emphasise the fact that the cooling speed used for this test was only marginally satisfactory.

The pearlite morphology of the specimens also varied, but this effect was not considered to be of sufficient proportion to be significant. Thus, although the continuously-cooled specimens generally contained more lamellar pearlite, the pearlite in nearly all the specimens consisted of a mixture of the lamellar and spheroidal forms.

The mechanical properties of the specimens are given in Table IV.

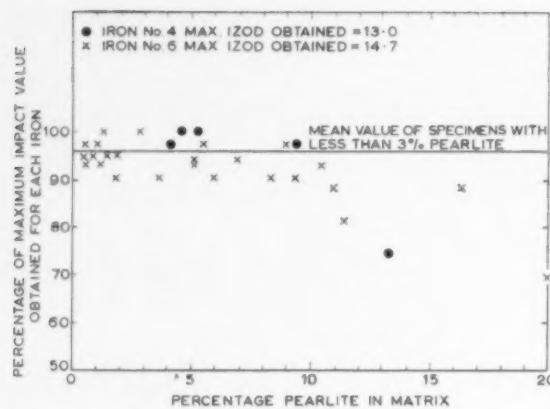


Fig. 9.—Effect of residual pearlite content on the notched impact values of S.G. iron.

It is therefore concluded that tensile ductility will be unaffected until the pearlite content exceeds 10%, but that notched room-temperature Izod values will be slightly impaired if pearlite is present in excess of 8-10%.

### Discussion

The results of this work show that it is possible to effect quite rapid ferritisation by controlled-cooling, especially in irons of compositions similar to that of Iron No. 1. Ferritisation by controlled-cooling will be considerably more rapid than that resulting from the two-stage process, but its superiority over three-stage processes will depend upon the amount of pearlite formed during the intermediate stage of the latter. Thus, if the furnace available for the treatment has a high natural cooling rate, then, as the castings pass through the eutectoid range a large part of the austenite will transform to pearlite. In these circumstances it would be better to use the controlled-cooling method, thereby ensuring that the predominant reaction is the ferritisation of austenite.

In practice, however, probably most annealing furnaces cool slowly over the important temperature range (800°-700° C.) and, unless the iron being treated contains potent amounts of pearlite stabilisers, only a small proportion of pearlite will form. In these cases this

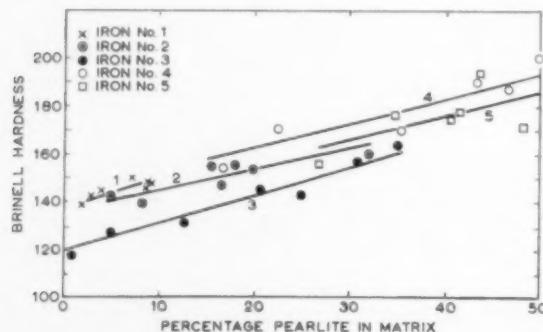


Fig. 11.—Effect of residual pearlite content on the hardness of S.G. iron.

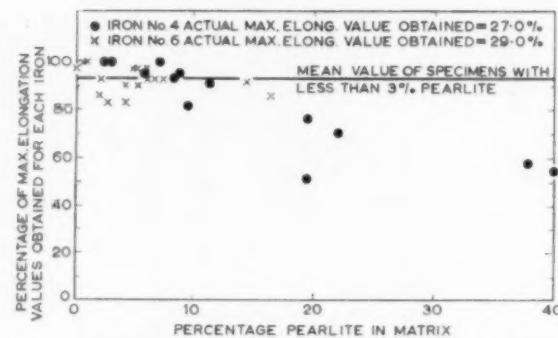


Fig. 10.—Effect of residual pearlite content on the tensile elongation values of S.G. iron

pearlite may be easily reduced by isothermal treatment, and, from this aspect at least, the controlled-cooling method will not be particularly advantageous.

Unlike the pearlite content, the hardness values at which deviation from the best results will occur will not have general significance, because they will obviously vary with the composition of the iron. The relationship between the percentage of pearlite and the hardness of the various irons is shown in Fig. 11. It should be noted that as only a small change in hardness occurs over the critical pearlite ranges careful measurements are necessary.

The conclusions reached concerning the amount of pearlite tolerable in highly ductile S.G. iron are in general accord with those of other workers. Thus, data presented by Rehder<sup>10</sup> and Eckel<sup>7</sup> suggest that at least 10% of pearlite can be present in annealed S.G. iron with little adverse effect on tensile elongation. In studying the effect of pearlite on the impact value Carr and Steven<sup>11</sup> did not detect deleterious effects below 10% pearlite, whilst Sandoz, Bishop and Pellini<sup>12</sup> came to a similar conclusion with regard to the effect of residual pearlite on the impact-transition temperature. Although in the present investigation the impact-transition temperatures of the irons were not determined, it is probably

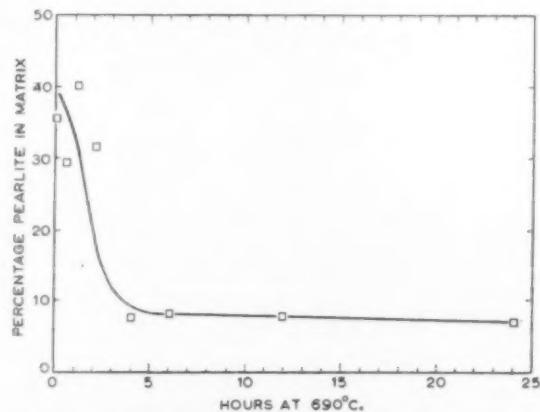


Fig. 12.—Effect of treatment at 690° C. on the amount of pearlite retained in Iron No. 5 after austenitising at 900° C. and cooling from 800° C. at 50° C./hr.

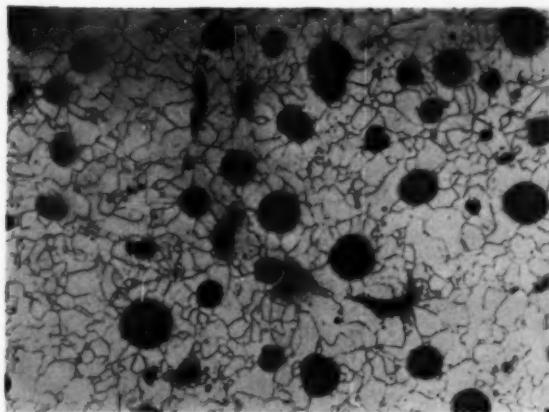


Fig. 13.—S.G. cast iron containing 3% pearlite. Etched  $\times 125$

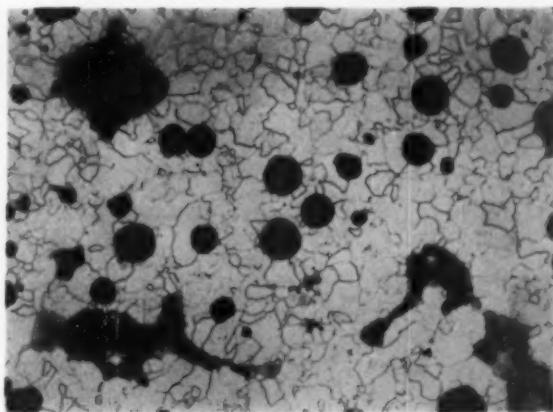


Fig. 14.—S.G. cast iron containing 13% pearlite. Etched  $\times 125$

significant that changes in the transition temperature can be related to an increase of hardness, and it is clear that no significant rise in hardness is found with 10% of pearlite present (Fig. 11).

To obtain high tensile ductility and impact values in irons which are inherently difficult to ferritise, it is probably preferable in any circumstances to use the three-stage process. Results from the treatment of Iron No. 5 are depicted in Fig. 12. These show that, as with the controlled-cooling method, even a prolonged treatment of this iron results in a marginal pearlite content. However, if the three-stage process is used the residual pearlite will be completely spheroidised and will therefore have the minimum adverse effect on ductility.

In Figs. 13 and 14 are shown the appearance of irons containing 3 and 13% pearlite, respectively, and slightly impaired properties would be anticipated from the latter.

### Conclusions

(1) From the results of the tests described it may be concluded that S.G. iron can be ferritised by continuous-cooling at controlled cooling speeds. The speed permissible will depend primarily upon the composition of the iron, and results for typical S.G. irons are given.

(2) Assuming a base iron of reasonable purity and a silicon content of 2.0-2.2%, it is suggested that the following approximate speeds of cooling through the critical range should, respectively, be suitable for irons of the compositions indicated below:—

| Iron                             | Maximum Rate<br>of Cooling<br>Over the<br>Critical Range :<br>800°-650° C.<br>(° C./hour) | Three-stage<br>process<br>should be<br>applied. |     |
|----------------------------------|---|---|-----|
|                                  |   | (a)   | (b) |
| (a) Nickel-free low manganese    | (Mn 0.10%)  | 80  |     |
| (b) Nickel-free medium manganese | (Mn 0.30%)  | 60  |     |
| (c) Nickel-free high manganese   | (Mn 0.50%)  | 30  |     |
| (d) 1.0% nickel low manganese    | (Mn 0.10%)  | 50  |     |
| (e) 1.0% nickel medium manganese | (Mn 0.30%)  | 30  |     |
| (f) 1.0% nickel high manganese   | (Mn 0.50%)  | 10  |     |
| (g) 1.5% nickel low manganese    | (Mn 0.10%)  | 30  |     |
| (h) 1.5% nickel medium manganese | (Mn 0.30%)  | 10  |     |
| (i) 1.5% nickel high manganese   | (Mn 0.50%)  |   |     |

(3) When using the controlled-cooling method, fast cooling from the austenitising temperature may be employed to 800° C. or slightly below, and it is unnecessary to cool at a controlled rate below about 700° C.

(4) It has been shown that the mechanical properties of continuously-cooled irons containing about 5% residual pearlite are quite satisfactory, and that somewhat larger proportions of pearlite may be present without impairment of tensile ductility or shock-resistance. Thus the presence of 10% residual pearlite will have no adverse effect on the tensile elongation values, whilst the critical pearlite content from the aspect of impact resistance, is suggested as 8-10%, depending upon the pearlite morphology.

(5) The circumstances under which it may be advantageous to use the controlled-cooling method have been outlined. It is suggested, however, that with certain irons it is clearly desirable to use a process which consists of furnace cooling from austenitisation, followed by isothermal sub-critical treatment.

### Acknowledgment

The author acknowledges his indebtedness to The International Nickel Co. (Mond), Ltd., for permission to publish this paper.

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# New Precision Orbital Welding Method

## Application to Bent Tube Assemblies

**A**SOLUTION to a major problem associated with the fabrication of complex tube assemblies—the bending to close limits and the subsequent welding of end fittings and other components to the fully-formed tube shape—has been provided by Palmer Aero Products, Ltd., a subsidiary company of B.T.R. Industries, Ltd., with the development of what is claimed to be a unique precision orbital welding method.

Welding is desirable for this purpose as it is generally recognised that it gives greater fatigue life and greater corrosion resistance (because it is homogeneous), and withstands higher temperatures when compared with conventional brazing techniques. However, welding a bent tube of small diameter and gauge thickness normally connotes a manual operation. Due to the awkward tube shape and small diameter, manual welding usually results in an uneven bead contour of large dimensions, marked at frequent intervals with "stop and start" craters. This type of deposit usually gives rise to a great deal of transverse stressing due to its size and is a focal point for stress patterns. This could prove a serious shortcoming in tubes subject to cyclic temperature, pulse pressure and high frequency vibration conditions. Furthermore, the uneven contour of the penetration bead may cause undesirable turbulence, and a change from one tube assembly to another may result



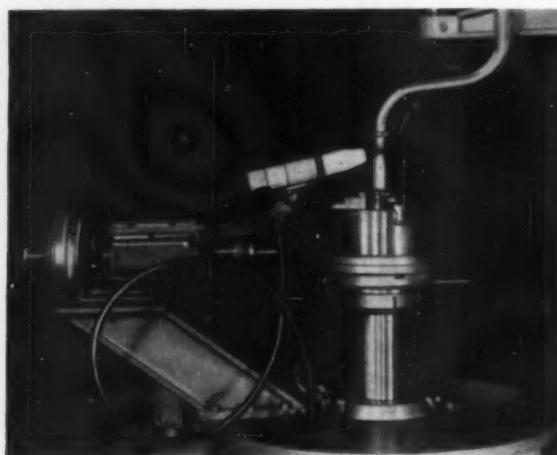
General view of the complete equipment.

in serious changes in rates of flow and frictional losses. From this it appears that in order to achieve a weld of uniform penetration and crown dimensions, and to achieve volumetric consistency from one tube assembly to the next, some automatic welding process is inevitable.

Usually, bending to close limits involves the use of discard lengths of tube at each end in order to ensure that bends are correctly positioned and that no flattening of walls takes place in the bend area. The additional leverage provided by the discard lengths is essential to the preservation of the correct radius and to the dimensional accuracy usually demanded. These factors necessitate the bending operation being carried out before welding, and the design of any automatic process must take into account the impossibility of rotating the workpiece under a fixed welding head.

It is therefore apparent that a torch must be developed which will orbit the pre-formed tube assembly. The new precision orbital welding method effects a butt weld between a fully-formed tube held in a fixed position and an end fitting positioned in correct relationship to it. The weld crown dimensions and penetration bead dimensions can be very accurately controlled, and such consistency can be obtained that designers are able to integrate the weld bead into design schemes. The degree of concentricity of weld crown and penetration bead with the tube is extremely high, and welds can be carried out to meet any desired X-ray standard.

The need for this type of accurately formed tube assembly with attached fittings abounds in the nuclear power industry, and in all industries concerned with handling corrosive materials at elevated temperatures. For example, *N.S. Savannah* has 43,000 ft. of small bore stainless steel tubing fabricated into lengths of 27 ft., and about 42 miles of similar tubing are used on the burst cartridge detection equipment at Calder Hall.



A close-up of the orbital welding head and holding device for the tube end fitting.

# Ductility in Internally Oxidised Silver Alloys

By J. A. Slade, B.A., and J. W. Martin, M.A., Ph.D.

(Department of Metallurgy University of Oxford)

*The intergranular brittleness associated with internally oxidised binary alloys of silver with aluminium is suppressed when small quantities of nickel or cobalt are present. The tensile properties and hardness of such alloys have been measured, and metallographic evidence suggests that the addition of the third element reduces the grain-boundary precipitate on internal oxidation. The necessary conditions for such suppression are briefly discussed.*

THE technique of internal oxidation has been employed to produce a dispersed phase in silver. Alloys suitable for this treatment consist of a dilute solid solution of a base metal in a more noble metal; the alloy is heated under oxidising conditions, so that oxygen diffuses into it and produces a dispersion of the oxide of the base metal in a matrix of the noble metal.

The effects of internal oxidation on mechanical properties have been briefly summarised in a previous paper by Seehofer and Martin.<sup>1</sup> Plastic deformation usually leads to intergranular fracture of these materials (see, for example, references 2, 3) due to oxide being concentrated in the grain boundaries as well as dispersed within each crystal, whereas internally oxidised single crystals behave in a ductile manner.<sup>1</sup>

The present study was prompted by results quoted without comment by various workers<sup>4, 5</sup> that, whereas an internally oxidised binary alloy of silver is hardened but embrittled, a ternary alloy of silver containing nickel as well as the base metal exhibits hardening, yet has appreciable ductility in a tensile test. It was decided to repeat this work and to study metallographically the differences between the binary and ternary alloys, with a view to accounting for the phenomenon.

## Experimental

### Preparation of Alloys

The composition of the alloys prepared is given in Table I. Silver of 99.99% purity was used in their preparation, and a number of confirmatory analyses were carried out by Messrs. Johnson, Matthey & Co., Ltd.

### Heat Treatment

The rate of internal oxidation of the silver-aluminium alloy was determined by heating wire samples in a stream of air in a furnace for known lengths of time, and the rate determined metallographically, a parabolic law

TABLE I.—COMPOSITIONS OF ALLOYS STUDIED

| Alloy | Composition                      |
|-------|----------------------------------|
| Ag    | Unalloyed silver, 99.99% purity. |
| BN1   | Ag + 0.02% Ni                    |
| BA2   | Ag + 0.18% Al                    |
| BA3   | Ag + 0.14% Al                    |
| T1D   | Ag + 0.08% Al + 0.07% Ni         |
| T2C   | Ag + 0.16% Al + 0.15% Ni         |
| T3A   | Ag + 0.14% Al + 0.002% Ni        |
| TCo   | Ag + 0.17% Al + 0.15% Co         |

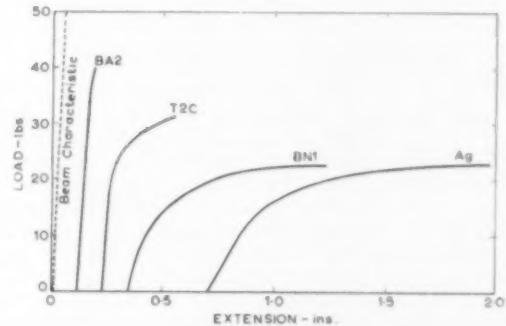


Fig. 1.—Load-extension curves for wire specimens: BA2 and T2C internally oxidised at 750°C; BN1 and Ag annealed at 750°C.

being evident. The temperature of oxidation employed was in all cases 750°C.

### Mechanical Testing

Microhardness indentations were made in some of the partially oxidised specimens, a Reichert microhardness indenter with a load of 10 g. being employed. The outer rim of internally oxidised alloy had an average hardness of 136 kg./sq.mm., whereas the unoxidised interior averaged 58.3 kg./sq. mm.

Two series of tensile tests were carried out, the first employing wire specimens, the second using specimens prepared from rolled strip. A Hounsfield Tensometer was used in each case. Before internal oxidation the specimens were annealed in argon (at the oxidation temperature) in order to stabilise the grain size. The oxidation treatment was carried out for such a time that the entire cross-section of the test-piece was internally oxidised.

Typical results of tests on wire specimens (0.036 in. diameter and 2 in. gauge length) are shown in Fig. 1, and some results are set out in Table II.

### Metallography

An optical micrograph of a longitudinal section through a tensile fracture of an internally oxidised silver-0.18% aluminium alloy appears in Fig. 2. The section was unetched, and the intense grain boundary precipitate of alumina is apparent; the resulting intergranular nature

TABLE II.—TYPICAL MECHANICAL PROPERTIES

| Alloy                  | Condition           | Ultimate Tensile Stress ( $10^4$ lb./sq. in.) | Elongation (%) | Hardness (V.P.N.) |
|------------------------|---------------------|---|----------------|-------------------|
| <i>Wire Specimens</i>  |                     |   |                |                   |
| Ag                     | Annealed            | 2.26  | 55             | —                 |
| BN1                    | Annealed            | 2.35  | 55             | —                 |
| BA2                    | Internally oxidised | 2.90  | 5              | —                 |
| BA2                    | Internally oxidised | 3.93  | 2.5            | —                 |
| T2C                    | Internally oxidised | 2.94  | 15             | —                 |
| <i>Strip Specimens</i> |                     |   |                |                   |
| Ag                     | Annealed            | 2.29  | 68             | 33                |
| BA3                    | Internally oxidised | 4.0   | 0              | 97.8              |
| T1D                    | Internally oxidised | { 8.84  | 40.7           | 74.3              |
| T5A                    | Internally oxidised | { 8.31  | 39.1           | 76.3              |
| TU0                    | Internally oxidised | { 7.8   | 20             | 81.2              |
|                        |                     | { 4.6   | 24.6           | 100               |

of the fracture-path is also evident. In all binary silver-aluminium alloys the precipitate was seen at high magnification, whether attack polished or completely unetched. Similar sections of fractured silver-aluminium-nickel oxidised specimens revealed no grain boundary precipitate, and the fracture was ductile in character and transgranular in path. Silver-aluminium-cobalt alloys were similar to these in their behaviour, although faint traces of grain boundary precipitate could be discerned at high magnifications.

The fracture surfaces obtained in the tensile tests on the above specimens were compared by obtaining two-stage replicas of their topography. Thick (0.03 in.) cellulose acetate was used for the first stage of replication, as it could be readily stripped from the fracture surface. The thickness reduced the possibility of introducing artificial strains during mechanical stripping. The second stage was the deposition of carbon; the plastic was then dissolved in acetone, leaving the carbon film. Gold/palladium shadowing was employed to improve the contrast, and the replicas were examined in



Fig. 2.—Silver-0.18% aluminium alloy, internally oxidised at 750°C. Section through tensile fracture, unetched.  $\times 500$

the electron microscope. A typical fractograph from an intergranularly brittle oxidised silver-aluminium alloy appears in Fig. 3, and Fig. 4 is the type of surface observed in the case of the more ductile ternary alloys.

### Discussion

The results of the mechanical tests are in agreement with those published by Schwarzkopf and Grant,<sup>5</sup> and the metallographic evidence suggests that the role of the nickel was to reduce the precipitation of oxide in the grain boundaries. This reasoning is supported by the work of Seeböhm and Martin,<sup>1</sup> who prepared binary alloys with low-angle boundaries. This reduced the degree of segregation and hence the intensity of precipitation; thus ductility was seen to be improved. The substitution of cobalt for nickel in silver-0.15% aluminium alloy was equally effective in removing intergranular



Fig. 3.—Silver-0.14% aluminium alloy, internally oxidised at 750°C. Electron micrograph of gold-palladium-shadowed two-stage carbon replica of the fracture surface.  $\times 10,000$

brittleness. Cobalt and nickel are of the same atomic size, and cobalt is even less soluble than nickel in silver.<sup>6</sup> The fractographs of Figs. 3 and 4 support this interpretation. The relatively smooth areas of Fig. 3 are presumably the surfaces of grains exposed, whereas the cusp-like markings of Fig. 4 are generally regarded as characteristic of a fracture of a ductile matrix containing hard inclusions.<sup>7</sup>

The Vickers D.P.H. number of the oxidised binary silver-aluminium alloys was greater than that of the oxidised ternary silver-aluminium-nickel. This may be interpreted by regarding deformation as being easier in the latter structures because of the suppression of the grain boundary precipitate. This suppression can be understood as the nickel competing favourably with

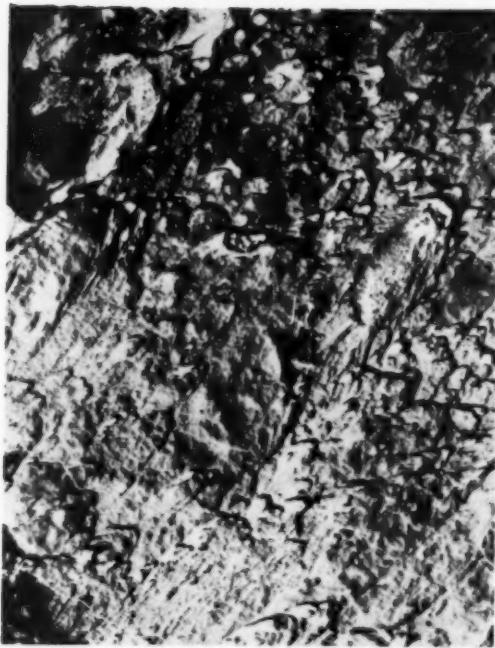


Fig. 4.—Silver 0.15%, cobalt 0.17% aluminium alloy, internally oxidised at 750 °C. Electron micrograph of gold-palladium-shadowed two-stage carbon replica of the fracture surface.  $\times 10,000$

aluminium for grain boundary sites, so that when the material is internally oxidised there is not the great concentration of aluminium in the boundary to give a dense alumina precipitate there.

Cahn<sup>8</sup> has stated that the concentration of a solute in a boundary is independent of the concentration within the grain, and he agrees with Eborall<sup>9</sup> that the degree of segregation is affected by size factors, but considers solubility a better guide because it expresses the combined effects of elastic and electronic factors. The influence of these respective factors can now be considered in the competition of nickel and aluminium for the grain boundaries of silver. Nickel is only very sparingly soluble in silver—about 0.1% at 950 °C., and only 0.01% at 400 °C.<sup>6</sup> The excess nickel, being insoluble in the grain will tend to concentrate in the boundary. Aluminium, however, has an appreciable solubility and forms electron compounds with silver. The atomic radius of aluminium (1.43 Å) is similar to that of silver (1.44 Å), whereas nickel is much smaller (1.25 Å) and so is likely to be accommodated more readily in the distorted grain boundary region.

In conclusion, therefore, one may suggest that there are four conditions to be fulfilled by the third element added to internally oxidisable alloys to reduce grain boundary oxide precipitates:

- (1) It must be only sparingly soluble in the matrix.
- (2) There must be a greater difference in atomic size between that of the parent metal and the third element, and the parent metal and the base metal.
- (3) It must be relatively noble compared with the

other solute metal, so that it is unaffected by the internal oxidation process.

- (4) It must not itself impair the mechanical properties of the resulting alloy.

#### Acknowledgments

The authors are grateful for laboratory accommodation made available by Professor W. Hume-Rothery O.B.E., F.R.S., Wolfson Professor of Metallurgy in the University of Oxford, and also acknowledge a grant from Messrs. Imperial Chemical Industries, Ltd., towards the cost of chemical analyses of the alloys.

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#### Oxyplane for New Steel Works

AN Oxyplane machine has been installed in the new South Works of the South Durham Steel and Iron Co., Ltd., West Hartlepool. The machine, installed by British Oxygen to trim plates coming off the new rolling mill, has a track length of almost 100 ft. Using propane as the fuel gas, the Oxyplane can trim plates up to 97½ ft. long and 12½ ft. wide, between ½ in. and 4 in. thick, and gives a square edge, bevel, double bevel, or double bevel and nose finish. Cutting speeds of up to 50 ft. an hour through 2 in. thick steel have been achieved. The Oxyplane works about four times faster than the portable cutting machines normally used in steelworks.

#### Electric Steel Makers' Guild

At the fifth annual meeting of the Electric Steel Makers' Guild, held at the Rowton Hall Hotel, near Chester, Mr. W. KEEN (Jessop-Saville, Ltd.) was elected president, Mr. R. S. Howe (Steel, Peech & Tozer) vice-president, and Mr. F. CRANKSHAW (K & L Steelfounders and Engineers, Ltd.) secretary/treasurer. After the meeting, members visited Brymbo Steel Works, Ltd., where, in the course of a tour of the melting shop, they saw the Brymbo oxygen-electric process in operation. Members were afterwards entertained to lunch by the company. At the Guild's annual dinner Mr. T. H. MEADOWS (director and secretary) and Mr. A. T. Harris (director and chief engineer) of Brymbo were the principal guests.

#### Beryllium Monitor U.S. Success

AFTER being shown at the American Industrial Hygiene Convention at Detroit, where it proved to be much superior to other types of equipment for the detection of beryllium particles in the atmosphere, the beryllium monitor made by Winston Electronics, Ltd., under licence from the United Kingdom Atomic Energy Authority, was taken to the United States Atomic Energy Corporation in New York, where it was demonstrated and given two exhaustive trials. As a result the instrument has been re-engineered to meet the special atmospheric requirements and high humidities in which it is required to work by the United States Atomic Energy Corporation.

## Vacuum Casting of Steel

After two years of trials on the vacuum casting of steel, involving the production of several thousand ingot tons, the Steel, Peech and Tozer branch of The United Steel Cos., Ltd., is believed to be the first company in this country to make steel produced by this method commercially available. The object of vacuum casting—or stream degassing, as it is sometimes called—is primarily to remove hydrogen from the steel. This can be achieved by suitable heat treatment of solid steel, but in the case of comparatively large section sizes, such as medium and heavy forgings, the heat treatment necessary to remove hydrogen by diffusion may take several hundred hours to complete. By casting the steel in a vacuum, however, hydrogen removal can be accomplished in a matter of minutes.

All hydrogen levels after vacuum casting are sufficiently low to eliminate completely the need for such normal hydrogen precautions as the slow cooling of rolled blooms or the extended heat treatment of large forgings. The oxygen content is appreciably lower than on air-cast samples, although the nitrogen content of the steel is not greatly affected by vacuum casting, only about 15% being removed on average. Carbon content tends to decrease by one or two points and sulphur is lowered by an average of 0.001-0.002%. Alloying elements remain unchanged, and this includes manganese.

Mechanical tests have shown that the properties throughout large forgings made from vacuum cast steel are more regular, and that the ductility, in particular, is good. Transverse properties are also improved. Impact properties are good and no significant difference in transition temperature has been found.

### Steel, Peech & Tozer Equipment

The equipment employed for vacuum casting at Steel, Peech and Tozer consists essentially of a cylindrical casing of  $\frac{1}{2}$  in. thick welded steel plates. This 11 ft. diameter chamber is fitted with a removable lid having water-cooled flanges, the vacuum seal being effected by a neoprene rubber gasket in the flange. The sequence of operations begins with the positioning of ingot moulds in the vacuum chamber. The lid is then secured and the chamber is evacuated by steam ejector pumps to 0.5 mm. of mercury. A ladle of molten steel is brought by overhead crane to the vacuum chamber and the steel is teemed through a small opening in the lid at a controlled rate. As the steel enters the chamber, it is broken up into a fine spray due to the explosive release of gasses, caused by the vacuum. It is this characteristic which enables very rapid degasification to take place, the process continuing at a slower rate when the steel is in the ingot mould. On completion of teeming, the vacuum is gradually released and the ingot allowed to cool in the normal way.

At the present time, steel for vacuum casting is being melted in a 7-ton electric arc furnace at Steel, Peech and Tozer. As an integral part of the company's £10 million scheme to replace all its open hearth furnaces with six 110-ton capacity electric arc furnaces, however, it is planned to incorporate facilities which will permit the vacuum casting of the very much larger tonnages of steel produced in these furnaces.

For a wide range of applications, steel produced by normal casting techniques is entirely suitable with regard to the properties for which it is specified. The particular merits of vacuum cast steel are in those cases—such as large forgings—where the elimination of prolonged heat treatment cycles (necessary to ensure freedom from hairline cracks) results in much shorter delivery dates; or where, in addition to hydrogen, the removal of oxides and nitrides may lead to a cleaner steel with improved properties.

### Heat Transfer Between Metals in Contact

BASIC information has been obtained on some of the factors which affect the transfer of heat between two machined metal surfaces. The transfer of heat between solid surfaces is widespread, for example, between fuel rods and cans which are in static contact in a nuclear reactor, and between the riveted components of an aircraft subjected to aerodynamic heating.

Measurements of the heat transfer between machined metal contacts have been made with brass and aluminium surfaces of four different values of roughness in contact with steel and brass surfaces of two different roughnesses. Contact pressure was varied between 14 and 11,400 lb./sq. in. It was found that most of the heat was transferred by surface contact and conduction through the intervening fluid. The fluid conductance was found, as expected, to be directly proportional to the thermal conductivity of the fluid and inversely proportional to an 'effective fluid film thickness'; the way in which the effective fluid film thickness depended on surface roughness was determined.

The conductance through surface contact was dependent on applied pressure, surface hardness, surface finish, and the thermal conductivities of the solids, and the relation between these factors was obtained. A theoretical expression for the surface contact conductance has been developed, based on the resistance to heat flow associated with a constriction. It gave good agreement with the experimental results when the variation of metal hardness with load in the micro-hardness region was taken into account.

This work was carried out for the National Engineering Laboratory at the Imperial College of Science and Technology, University of London. Further information is given in: *NEL Report Heat 182* (Thermal and electrical conductance of machined metal contacts, by L. C. Laming) available from the Laboratory at East Kilbride, Glasgow.

### Kite-Mark Indicators

BRITISH INDICATORS (SALES), LTD., St. Albans, Herts., have been granted a licence by the British Standards Institution to apply the Kite-Mark to their range of dial gauges for linear measurement manufactured in accordance with B.S.907. When this certification mark appears on a product it is an assurance that the product is "up to standard." The mark is used on any product only under licence after B.S.I. has satisfied itself, by inspection of the manufacturer's production processes and works control and by testing products, that the product is consistently manufactured in accordance with the relevant British Standard. It indicates not mere testing and approval of a submitted sample but continuous supervision with unannounced visits of inspection and check-testing of samples bought on the open market.

# National Physical Laboratory Open Day

## Research Progress in Metallurgy Division

A feature of general rather than technical interest at the National Physical Laboratory's 1961 Open Day was the new Glazebrook Hall, converted from the former duplex wind tunnel to provide the Laboratory with a fully equipped lecture theatre to seat almost four hundred, a conference room, a reading room, and a restaurant, the latter serving also the adjacent National Chemical Laboratory and the Admiralty Research Laboratory.

The new premises meet the long-felt need for a larger lecture theatre than was formerly available to provide accommodation for the inter-divisional colloquia and lectures for N.P.L. staff, and for the international symposia, which have been held for some time at the rate of two a year, and which have brought to Teddington many of the leading figures in wide-ranging fields of work. The Laboratory has also won fame for the strength and variety of its social activities, which have flourished despite the inadequacy of accommodation and catering facilities provided by three large huts.

Completed early in 1921 the duplex wind tunnel played a major part in the development of aeronautical knowledge during the formative years of the aircraft industry. Probably the most exciting work carried out there was related to the development of successful aircraft for the Schneider Trophy contests in 1927, 1929 and 1931. Latterly it was used extensively for industrial aerodynamics, models tested including television towers, the dome for the Royal Observatory at Hurnstmonceaux, and the Jodrell Bank radio-telescope. Wood from the old tunnel has been used to panel the rear wall of the lecture theatre and upper gallery, thus maintaining a link with the past. Another has been preserved in the new name of the building, which has been called after Sir Richard Glazebrook, the first director. A memorial plaque to Sir Richard has been placed on the west wall of the assembly area.

### METALLURGICAL RESEARCH PROGRESS

#### High Temperature Materials

With the transfer of creep machines from the high temperature materials laboratory at Teddington to the National Engineering Laboratory at East Kilbride, and the associated transfer of materials, test pieces and records—some to East Kilbride and some to the Electrical Research Association at Leatherhead—the facilities at the N.P.L. have now to be reorganised for the new work that is planned, which will include a greater concentration upon new materials of very high melting point.

The work on display in the high temperature material field included a study of the factors influencing the creep rupture elongation of austenitic nickel-chromium and ferritic chrome-molybdenum steels. The ductility at fracture in creep tests on 18% chromium-12% nickel-1% niobium steel has been found to pass through a minimum for times of intermediate duration at 650° C. and 700° C. As low ductility is an undesirable feature

for superheater tube and steam-pipe applications, a metallurgical examination using a replica technique with the electron microscope is being carried out to see if a structural reason can be found for the minimum. This has shown that when minimum ductility occurs two structural changes have taken place in the steel. Niobium carbide (NbC) has been precipitated in the austenite matrix and chromium carbide ( $\text{Cr}_{23}\text{C}_6$ ) has formed at the grain boundaries. The latter would be expected to lead to chromium depletion in the grain boundary regions. In longer times, i.e. beyond the time required to produce minimum ductility,  $\sigma$  phase forms. This phase was found in all long-time tests in the temperature range 600°-700° C., but the ductility is not necessarily low when  $\sigma$  is present. Above 700° C., the  $\sigma$  phase produced in creep tests reaches a maximum proportion, and then appears to redissolve with the formation of a new phase having a higher chromium content with a composition corresponding to  $\text{FeCr}_2$ .

In the case of a 1% chromium-1% molybdenum steel for steam pipes, creep tests at 550° C. over a stress range of 8-25 tons/sq. in. have been carried to rupture with an indication of minimum ductility in 1,500-1,700



West front of new Glazebrook Hall.



Transmission electron micrograph of iron-carbon alloy (0.05 wt.-% carbon) quenched from 690° C. and aged 18 months at room temperature. The dendritic precipitate is iron carbide that has nucleated on stationary dislocations.

× 40,000

hours, as compared with the 2,000–2,500 hours in the case of a 1% molybdenum steel. Again, structural changes probably account for the minimum ductility, which has been shown to be associated with the precipitation of molybdenum carbide ( $Mo_2C$ ) in the ferritic matrix in the 1% molybdenum steel. In the case of the chrome-molybdenum steel,  $Mo_2C$  has not been identified in extraction replicas from rupture test specimens until the period of minimum ductility has passed. However, the  $Fe_3C$  initially present has changed to  $(FeCrMo)_{23}C_6$  type carbide by the time minimum ductility is reached.

In the course of an investigation of the possibilities of increasing the use of niobium in alloys for high temperature applications, extensive tests have confirmed that the resistance to both creep and relaxation of a 90–10 nickel-chromium alloy after various amounts of cold work is increased more by additions of niobium than by equal titanium additions. An alloy containing 2½% niobium and cold worked 30% has a creep resistance in the range 500°–650° C. similar to that of some well-established austenitic steels requiring complex heat treatments to develop their maximum creep strength. It is estimated that the niobium-bearing material would have a life of some years at 600° C. without recrystallising, and it is thought that alloys of this kind may have applications where resistance to repeated plastic deformation is part of the essential requirements.

A machine designed to measure the resistance of alloys to alternating strain under constant or fluctuating temperature has been used to test the suggestion that resistance to thermal fatigue is largely governed by the resistance of the material to alternating strain at the highest temperature of the thermal cycle. A series of tests on molybdenum-vanadium steel strongly supports the view and tests are being undertaken on other materials.

The machine has been modified by the introduction of resistance heating of the specimen, which has enabled the maximum temperature to be raised to 1,000° C., and a contract has been accepted for a study of the factors influencing the thermal fatigue resistance of nickel-chromium alloys, which are subjected to severe cycles of thermal stress in service in gas turbines.

In the earlier tests on steels at lower temperatures it was found that the resistance to alternating strain, when the temperature was fluctuated in phase with the applied strain cycle, was about the same as if the temperature were held constant at its maximum value. If this proves to be generally true it will be possible to determine the influence of metallurgical factors on thermal fatigue resistance by tests at constant temperature. A comparison of the behaviour at constant temperature and with the temperature fluctuating is now being made on Nimonic 90.

At room temperature, ductile metals are able to withstand a range of strain of about 2% for 1,000 cycles. The results of the earlier tests made on steel and those obtained so far on Nimonic 90 show that this value is much reduced at high temperatures, that is, these materials become much less resistant to repeated plastic deformation as the temperature is increased.

#### Brittle Fracture

In an investigation of a large crankshaft that broke in a brittle manner as a result of an accident, it was found that the fracture stress of the material, when it broke in a brittle manner at low temperature, was highly dependent on the size of the test piece. The yield stress, when, at high temperature, normal yielding preceded fracture, was practically independent of test-piece size. The differences between the factors that affect the yield stress of a component and those that affect its fracture stress, are thus many and important, particularly in connection with the very high tensile steels that are now being developed and their application in pressure vessels.

Measurements of the effect of strain rate on the mechanical properties of 4% silicon iron have shown that, whereas increase of strain rate raises the yield point of the material, it has no significant effect on the fracture stress of material breaking in a brittle manner at low temperature. This work has also shown this material to be very susceptible to variations of grain size, which again has a much greater influence on the fracture stress than on the yield point. The grain size effect in pure iron and a 5% molybdenum-iron alloy is less marked, differences also being found between samples of the same grain size produced by different methods. These experiences have led to a renewed interest in the relations between grain size, yield point and fracture stress.

The growing use of cold-formed steels lends interest to the discovery of a curious weakness displayed at low temperatures in the direction of compression in cold compressed pure iron—again unaccompanied by any significant yield stress change. The weakness begins to be removed on annealing at 500° C., being complete at 600° C., coincidentally with the removal of the work hardened structure. It is believed that the way in which the metal is deformed is of importance, but the shapes of the grains and grain boundaries and the distribution of inclusions are not vital factors in producing the weakness.

### Electron Microscopy

Until the technique of examining thin metal films under the electron microscope was developed, it was not possible to observe directly the form and distribution of dislocations in metals. The thin film technique has led to interesting and important results, and the method is proving to be particularly suitable for studying the precipitation of phases from solid solutions.

Plastic flow in metals takes place by the movement of dislocations, and where they can move easily, as in a pure metal, the metal is soft. It is believed that precipitate particles confer strength by acting as obstacles to dislocations and it is expected that many details of the interaction can now be studied. An investigation on iron-carbon alloys has shown that precipitation occurs predominately on dislocations and that at room temperature and 100°C., the precipitate is mainly epsilon carbide ( $Fe_{2.4}C$ ), a close-packed hexagonal phase growing on (100) planes in a  $\langle 100 \rangle$  direction. In an iron-nitrogen alloy the precipitate occurs to a large extent between dislocations and grows in (100) planes in the form of discs as the ordered body-centred cubic structure  $Fe_{16}N_2$ . Dislocations have been observed to wrap round these obstacles and this is believed to be responsible for the increased resistance to deformation. These studies are being extended to commercial steels and it is clear that the additional elements present in these can affect the precipitation processes.

To obtain high quality thin foil electron micrographs it is necessary to tilt the specimen critically. The tilting stage supplied with the microscope is too restricted in its action, so completely new stage has been designed and constructed. The stage should allow the direct determination of the Bergers vectors of dislocations, the determination of the crystallography of precipitates with respect to the parent matrix, and the facility for producing good quality stereographic pairs of micrographs.

To avoid any shift of the observable specimen field

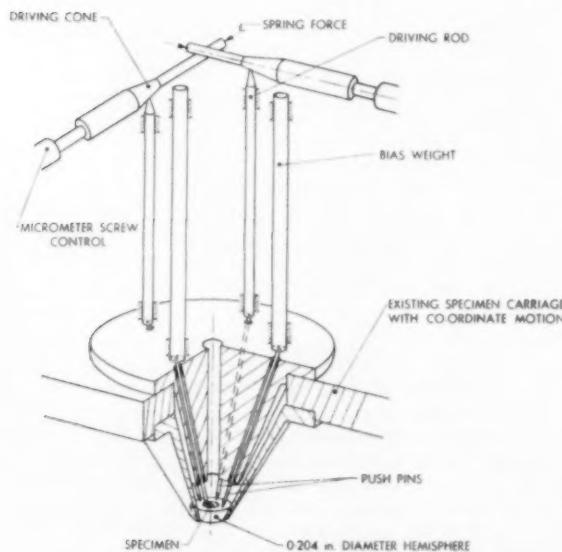
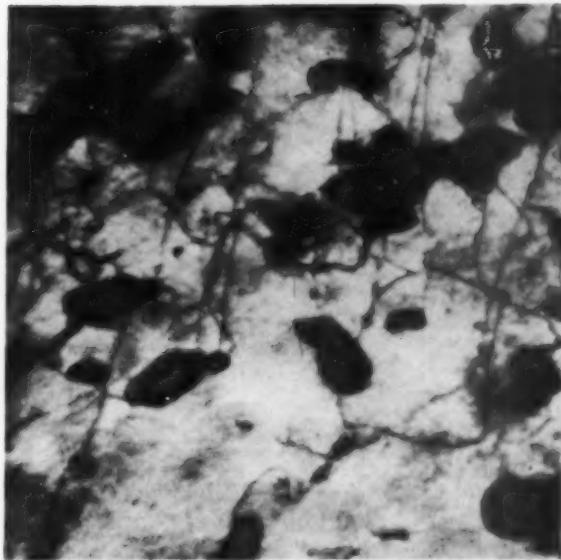


Diagram of the goniometer stage for the electron microscope.



Transmission electron micrograph of iron-nitrogen alloy (0.01 wt.%, nitrogen) quenched from 550°C. and aged 3 days at 100°C. and then lightly strained. The iron nitride precipitate particles can be seen to have acted as obstacles to moving dislocations.  $\times 40,000$

when a specimen is tilted, the design of the goniometer stage was based upon the simple kinematic system of locating a ball in an inverted cone, which provides a method in which specimens, mounted to intersect the ball centre, may be tilted without shake. In this application a hemisphere is used, tilting being effected by the action of four slender push pins angled at 15° which contact the diametric plane of the hemisphere. By tilting the hemisphere through approximately  $\pm 22^\circ$  about each of two horizontal axes at right angles to each other, a composite tilt of  $\pm 30^\circ$  is obtained. Each push pin makes contact at its upper end with the horizontal end face of an actuating rod. The horizontal end faces of each of the rods allow free movement of the specimen carriage without interference with the specimen tilt. The upper ends of each of two of the actuating rods make contact with a conical portion of individual horizontally disposed shafts, each of which is forced by an axial spring to follow the movement of the spindle of a commercial micrometer. A dead-weight is placed on the upper end of each of the other two rods in a manner which, although avoiding undesirable excessive loading on the hemisphere, ensures that the tilting movement of the hemisphere follows the micrometer actuated rod when the latter is retracted.

### Brittleness in Titanium-Aluminium Alloys

The tensile properties of a number of titanium-aluminium alloys at room temperature and  $-196^\circ C$ . have been determined in order to follow the decrease in ductility with increase in aluminium content up to about 10%. It is known that brittleness develops with time during heat treatment at about  $600^\circ C$ . when the aluminium content exceeds about 8%. Alloys of this composition are also difficult to hot work, so that considerable delay has been experienced in obtaining an extruded 10%

aluminium alloy. No brittleness has been produced in alloys containing up to 7½% aluminium on heat treating at 600° C., but the brittleness is well developed in the alloy containing 10% of aluminium. The embrittled alloy decreases in tensile strength as the temperature falls from room temperature to -196° C. and the elongation and reduction of area at fracture decrease to zero. This type of behaviour is of great interest, since it has hitherto been observed principally in iron alloys which have been rendered susceptible to intercrystalline embrittlement by the introduction of an impurity.

#### Chemical Thermodynamics of Alloys

The science of "chemical thermodynamics" is concerned with the measurement of heat effects of chemical reactions as well as of electrochemical reactions and of equilibria between a solid and the vapour it forms. The numerical information obtained by these measurements is condensed into tables listing the so-called standard heats of formation, entropies and specific heats of all pure substances such as metals, alloys, oxides, nitrides, sulphides, etc. These tables can be used to assist other laboratory work along similar lines intended to achieve a deeper understanding of the nature of the atomic bonding in alloys and other inorganic substances.

The practical significance of thermochemical data lies in the fact that they can be used to calculate various equilibria attained or encountered in the production and refining of metals and alloys, in particular at high temperatures. The data make possible, for instance, the calculation of the extent to which a certain agent would reduce a certain ore to produce the metal, or the extent to which a liquid alloy would pick up impurities from a given refractory used as the container. Present industrial developments require metals of ever increasing purity, and here again thermochemical calculations help to assess the value of methods for the refining of these metals. Phase relationships in alloy systems, the knowledge of which is fundamental to all metallurgical work can also be calculated from thermochemical data.

The application of thermochemical calculations to industrial problems is still in its early stages, but gradual progress is being made, and the N.P.L. contributes to this progress by devising new methods for the determination of the required data, by collecting and assessing the information published by other workers, and by demonstrating their potential usefulness to industry. A new high temperature calorimeter has recently been constructed in which the heats of formation of solid, low-affinity alloys can be determined directly at temperatures between 1,000° and 1,250° C. The calorimeter is to be used first on alloys the free energies of which have been derived from equilibrium studies, because it has now become clear that the conventional evaluation of heats and entropies from the temperature coefficients of equilibrium data is very unreliable for solid alloys, and that a re-appraisal of many of the published thermochemical data is necessary in such cases.

#### High-Purity Metals

The decisive effect of certain impurities on the mechanical and physical properties of metals has become increasingly recognised during the past decade, and a study of this subject necessitates extremely sensitive and accurate methods of analysis. No single analytical

technique is universally satisfactory for such work, and the chemistry section is equipped for determinations by mass spectroscopy, by emission spectroscopy and by neutron activation methods. The importance of control of the impurity content during preparation of metals and alloys emphasises the necessity for methods of melting in which rigid control can be exercised. Three methods were on show to visitors: (a) high frequency melting in controlled atmospheres, e.g. in hydrogen or vacuum; (b) electric-arc melting of reactive metals using consumable or non-consumable electrodes; and (c) electron beam floating zone melting.

An approach has been made to the Warren Spring Laboratory to undertake work on the electrolytic method for the production of high purity iron on a production basis. Meanwhile, experiments have been carried out to investigate the possibility of growing ingots of high purity iron by the Czochralski technique in an induction-heated vacuum furnace. Several 3-5 lb. ingots have been obtained, and mass spectrometric analyses indicate a reduction in their tin, antimony, sulphur and phosphorus contents compared with those of the starting material: a sulphur content of below 5 p.p.m. was recorded on one occasion. The technical difficulty of holding a 50 lb. melt at a constant temperature a few degrees above its solidification point has not yet been solved.

#### AEI's First Scientific Film

"ELECTRON MICROSCOPY" is the title of the first scientific film to be made by Associated Electrical Industries, Ltd. Its subject is of immense importance in modern science, since the facts that a beam of electrons has a much higher resolving power than light and can be focused with electro-magnetic coils resulted in the development of a microscope which can observe objects much too small for even the most powerful light microscope. It even, with the image intensifier, opens up the possibility of observing individual atoms. The history of microscopy and the principles of this modern method are explained. The film also shows the construction and operation of the EM 6 microscope and its use with the image intensifier, the preparation of specimens, and a number of its manifold uses in research and industry.

"Electron Microscopy" is designed for two primary types of audience: one consisting of scientists and technicians in fields other than electron microscopy, and the other students, undergraduates and graduates. The running time of this Eastmancolour film is 23 minutes, and 16 mm. and 35 mm. copies are available on loan from the AEI Film library, Associated Electrical Industries, Ltd., Crown House, Aldwych, London, W.C.2.

#### Correction Annual Furnace Survey

In the description of the Leeds Northrup Steam Homo Furnace which appeared on page 298 of the June issue of METALLURGIA it was stated that during this steam treatment a very thin film of oil-bearing zinc oxide is absorbed into the metal. This was an error. The micro thin film (0.0001 in.) formed on the work is an oil-bearing iron oxide ( $Fe_3O_4$ ) which it is claimed gives greatly prolonged life to all cutting tools, gears and bearing surfaces.

# Making, Working and Testing of Steel

## B.I.S.R.A. Research on Show at Sheffield

BUILT over the period 1950 to 1953, the Sheffield research station of B.I.S.R.A. (The British Iron and Steel Research Association) was opened by H.R.H. the Duke of Edinburgh in November 1953, and since that time has played a worthy part in the development of the iron and steel industry of the United Kingdom. The Hoyle Street laboratories house three of B.I.S.R.A.'s five divisions—steelmaking, mechanical working and metallurgy—together with the steel user section of the development and information services. Other B.I.S.R.A. laboratories are located in London, Swansea and Normanby (near Middlesbrough) and open days are held at the various centres from time to time. This year it was Sheffield's turn, and a most interesting display was put on there on the 15th and 16th June. To deal fully with all the demonstrations and exhibits would take much more space than is available : it will be necessary, therefore, to confine the following account to some of the highlights in the various fields.

### Steelmaking

#### *Spray Refining Blast Furnace Metal*

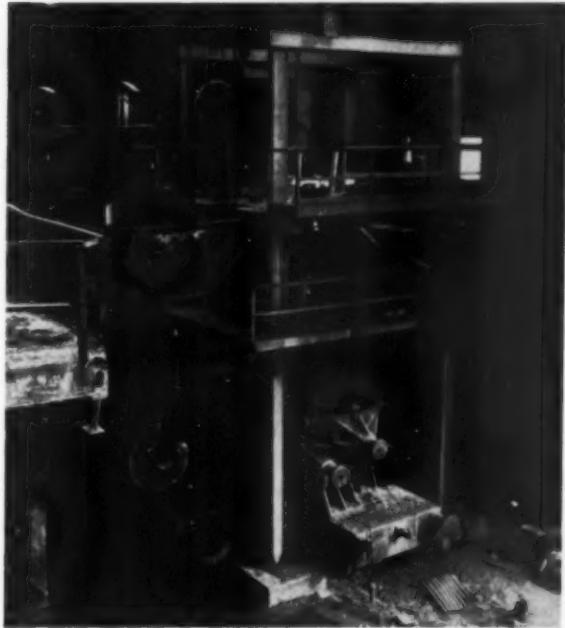
If a stream of molten blast furnace metal is atomised by a suitable arrangement of oxygen jets, there is rapid oxidation and refining of the metal. When fully developed such spray refining should be a simple and inexpensive method for pretreating iron to make it suitable for finishing to steel in any type of furnace or converter at rates that match the requirement of the finishing process.

Following the promising results obtained from laboratory experiments on a 10 cwt. scale, a pilot plant has been designed with the help of the engineering staff of Richard Thomas and Baldwins, Ltd., Redbourn Division, and experiments are in progress at Redbourn. A throughput rate of 2.5 tons/min. has been attained, and batches of hot metal up to 16 tons have been treated at rates up to 2 tons/min. with oxygen flow rates ranging from 250-600 cu. ft./min. At low metal flow rates the metal can be adequately atomised and oxygen used very efficiently, and experiments are in progress to determine the form of atomising device that will ensure adequate utilisation of oxygen at high metal flow rates to give any desired degree of refining.

By means of this spray refining process, efficient and rapid desiliconisation is possible, with the simultaneous removal of manganese, chromium and vanadium. In addition, when powdered lime is injected with the oxidising gas it is possible to dephosphorise in the order of 25-30% at the 1.3-1.4% levels, loss of iron and manganese to the slag also being reduced.

#### *Degassing of Molten Steel*

A pilot vacuum degassing plant for the continuous treatment of molten steel has been installed in the Sheffield laboratories. In this plant a stream of molten



*Courtesy of Richard Thomas & Baldwins, Ltd.*

**Pilot plant for spray-refining hot metal.** Molten iron from a tundish on the upper platform is atomised as it passes through a ring manifold which provides a number of high velocity oxygen jets and which is situated on top of the fume-extracting hood. The refined metal falls into the transfer ladle below.

steel flows upwards under a ferrostatic head into an evacuated chamber where the spray of steel is degassed. It is then discharged directly down a barometric leg to a tundish below the vacuum chamber. By this process it should be possible to treat large quantities of steel in a comparatively small vacuum chamber.

A substantial advance has been made in the use of carrier gas techniques for removing hydrogen from molten steel. It has been found that by directing a jet of the inert gas on to the surface of the molten metal the hydrogen content is reduced to low level in a short time. In the laboratory induction furnace the hydrogen content of 80 lb. melts has been reduced from 8 p.p.m. to 1 p.p.m. in about ten minutes. The method has been called "jet degassing."

Previous methods of gas flushing in which the gas was bubbled through the metal have proved incapable of reducing the gas content to less than 3 p.p.m. The reason for this seems to be that when gas is injected below the surface of the metal the slag and metal are intimately mixed and hydrogen is continuously transferred through the slag into the metal. When the jet is directed on to the surface the slag is swept to one side

and plays very little part in the reaction. Theoretical calculations suggest that 1 p.p.m. is the limit to which hydrogen can be reduced by jet degassing, but this should be adequate for most commercial purposes.

#### *Electric Steelmaking*

After successful trials on the B.I.S.R.A. experimental furnace, and on a five-ton electric arc production furnace, a member firm has built and satisfactorily worked its own model of the B.I.S.R.A. automatic power input controller (A.P.I.C.), and a further controller has been installed for trial on a 30 ton furnace at another member's works. The controller, which limits the high voltage period to pre-set times, has worked satisfactorily and has achieved a reduction of electrode consumption of 2 lb./ton of steel and a reduction of electricity consumption of 25–30 kWh./ton. A simple form of A.P.I.C. has been designed for use with modern furnaces having on-load tap changing and tap pre-selector switchgear: this equipment is now being manufactured by four United Kingdom firms.

Rapid desulphurisation of steel to low levels has been achieved by injecting powdered desulphurising reagents into the metal bath of a basic electric arc furnace. Works trials have shown that similar results can be obtained on production furnaces, but the correct operating procedure for furnaces of different sizes needs to be established.

#### *Continuous Casting*

A demonstration which attracted considerable attention was of continuous casting, a method of producing ingots, in various sections, which are so long in relation to their other dimensions as to have a negligible amount of top and bottom wastage. Because of the better yield, ingots may be cast nearer to the semi-finished billet dimensions than is customary in the conventional ingot process. The smaller section results in a shorter time for solidification, although the product is still an ingot requiring to be worked to break down the as-cast structure and develop the properties of a wrought metal, a reduction of 85% being necessary to ensure consistently high mechanical properties.

The macro-structure of the ingots usually includes an outer chill zone, a columnar zone and a central equiaxed crystal zone with central porosity which is removed in hot working, as are sub-surface corner cracks sometimes produced if the section is distorted by asymmetric cooling. The recently developed method of casting three ingots simultaneously in a mould having connecting compartments has very little effect on the structure of the ingots: when this multiple ingot is split for rolling, the resulting individual ingots are identical with those cast in a single mould.

Analytical surveys have shown only very slight segregation of carbon, sulphur and phosphorus in the transverse and longitudinal directions, and segregation in continuously cast rimming steel slabs is less than that found in conventionally cast rimming steel slabs. The longer refractory path with tundish teeming as compared with conventional top pouring seems to have little adverse effect on non-metallic inclusion content, but the high preheat calls for a higher quality refractory than that for conventional casting pit hollow-ware.

Continuously cast ingot surfaces are generally very good provided the casting conditions are effectively controlled, and sections of 3 in. and 4 in. square ingots

have been upset-forged without any splitting of the undressed faces.

In recent years it has become clear that continuous casting is more suitable for bulk steel manufacture when large sections are cast, because the throughput increases with section size even though casting speeds decrease. There is a limit to the increase in size of square sections if the product is not to require cogging, but wide slabs may be cast at satisfactory production rates and yet be small enough to by-pass the cogging mill. Some work has been carried out at the Abbey Works of The Steel Company of Wales, in collaboration with the company, and 24 × 4 in. and 27 × 5 in. slabs have been cast and sectioned, one of the larger ones being successfully rolled into sheet. The commercial twin-strand plant for casting low carbon slabs up to 48 × 8 in. section at the Abbey Works, is based on the continuous casting system developed by B.I.S.R.A. and the Continuous Casting Co., Ltd., and will be the first plant in the world to operate commercially with converter steel.

### **Mechanical Working Operations**

#### *Forging Production*

The feasibility of making a forging automatically by programme has been demonstrated on the experimental forge in the metal working shop. Forging an ingot into a bar having three parts of different dimensions took 6 minutes as compared with an estimated 15–19 minutes on a conventional plant. Savings, both in time and in heating costs, are likely to be greater with more complicated forgings, and the next phase of the work will be to install what is virtually an industrial prototype programmer capable of making a range of forged shapes. Substantial improvements to the linked control systems of the manipulator and press are necessary to enable its performance to meet the programming requirements, and a comprehensive scheme for this has been worked out and is being developed jointly with two associate member firms.

Manual tool changing at the press, however expeditiously carried out, constitutes a substantial loss of useful forging time, and automatic methods of changing tools could be of definite advantage in enabling the best use to be made of programmed forgings of complicated shapes. An experimental automatic tool changer of the turntable type has been designed for use on the experimental press and more versatile types for industrial use are being considered.

While the present work on programmed forging is based on existing types of presses and manipulators, plant developments both here and abroad suggest that the time is opportune for an entirely fresh approach to the principles of design of a non-specialised forge, with particular emphasis on reducing the high cost of new installations. Design studies involving new principles of press design and methods of manipulation and transportation have been started.

The thermal efficiency of a batch-type forge furnace is influenced by a number of design and operational features, and tests carried out on a modern unit of this type have suggested a number of basic improvements that might well be applied to the design of forge furnaces in general. This work is of considerable importance because heating costs form such a large part of the costs of forging production. For the same reason a good deal

of interest has been shown in the application of radiant heat reflectors during forging. By this means the cooling rate is slowed down and more forging can be carried out per heat.

Cavities and porosity are present on the axis of ingots as a result of shrinkage, but research has shown that, if applied with certain rules, upset-forging will guarantee that all such cavities will be closed-up. The recommended procedure is now in use industrially.

#### *Manufacture of Rounds and Sections*

Rolling with only one roll driven has advantages in respect of cheapness and mechanical reliability, but there are difficulties in spinning the idle roll up to speed and one roll wears more quickly than the other. A theory of rolling under these conditions is being developed to help solve the problems. An unexpected prediction from this theory is that with single-roll drive torques can be up to 25% less than those needed with the normal system.

Reducing wear on the rolls in the finishing train of a rod or bar mill would improve not only productivity but also quality, because uneven pass wear causes distortion of the stock, and this disturbs the smooth operation of continuous mills and introduces defects into the product. Works trials of rolls fitted with rings having facings of hard-wearing materials are to be made.

The design of the experimental three-stand mill to be installed in the laboratory has been completed, and the plant is expected to be commissioned later this year. The stands are designed on the opposed cantilever principle and a pre-prototype stand of this design has been built and fitted with a drive for testing.

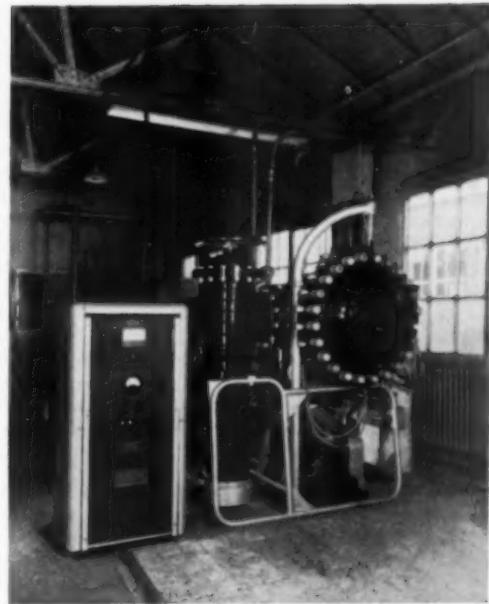
Satisfactory results have been obtained in the trial production of high-speed steel sections by hot extrusion of ingots, but there is a need for cheaper and simpler presses so that small quantities can be produced economically. This aspect is being actively studied, with the intention of installing a versatile press for the investigation of several new and promising methods of hot deformation.

#### *Wire-drawing*

In drawing high carbon steel wire it is necessary to ensure that excessive temperatures are avoided, because of their known effect in causing a deterioration in wire properties. Consequently the output of a machine is dependent on cooling efficiency. Up to 30% of the heat can be removed by external air blast, but beyond this point, the method is uneconomic.

Modifications in water cooling can, however, lead to substantial increases in drawing speed and wire output. In the B.I.S.R.A. system of cooling, the narrow gap between the drawing ring and the stationary inner cylinder of the capstan, ensures that a maximum area of capstan wall will be wetted by the cooling water passing through the gap. The narrower the gap the higher is the rate of heat transfer, because of the increased efficiency in causing turbulent flow. Additionally, the inner capstan walls are plated to prevent corrosion and a chemically treated water is used in a closed circuit cooling system. These precautions prevent excessive rusting and the silting up of both capstan and pipes by deposits present in untreated water.

Weld breakages in high carbon steel wire are a source of serious trouble in continuous wire-drawing mills, failure occurring most frequently in wire thinner than



**A small vacuum or pressure furnace.**

16 s.w.g. and in heavy gauge rod in the size range 3 s.w.g. and thicker. It has been shown that during welding of the more rigid rod, the main problem is end preparation and alignment, a difficulty which may be overcome by flash butt welding, where the end preparation is not so important because the flashing operation performs this task. With the thinner gauges, the difficulty of controlling the cooling rate results in the formation of a low-strength fine-grained band on each side of the weld, and a low temperature heat treatment cycle has been recommended to overcome this. Automatic control of these post-welding treatments on welding machines would greatly improve the consistency of the welds produced, and a survey of existing machines has been made with suggestions for improving their performance.

New equipment being introduced into wire mills will make it unnecessary for the operator to stop his machine after drawing 2-4 cwt. of wire to remove the coil from his machine, as coil weights of 2 tons are likely to become quite general in the near future. It will still be necessary for him to stop the machine from time to time to check the surface finish and diameter of the wire. The development of satisfactory means for continuous in-line inspection would eliminate the stoppages and reduce the amount of rejected material. As yet there are no instruments available for the continuous inspection of surface finish or the detection of score marks, but considerable advances have been made in automatic gauging techniques. B.I.S.R.A. has made a survey of the wire gauging instruments on the market, classifying them into contact and non-contact types. The former may be operated by mechanical, pneumatic or electrical means, the wire passing between rolls or stylus. Non-contact gauges (which are particularly advantageous for measuring fine wire, where a very good surface finish is often required) fall into three categories—electrical (capacity),

pneumatic, and the optical type using photo-electric cells.

#### *Manufacture of Sheet and Strip*

The removal by pickling of the edge scale on both narrow and wide mild steel strip may take three or four times as long as the centre scale, thus wasting time, acid and metal, as the centre is over-pickled. Mechanical scalebraking shortens the pickling time only marginally. Examination of narrow strip has shown that a film of  $Fe_2O_3$  is formed on the rolling scale near the edge during the slow cooling of the strip after coiling, and though this oxide has not been positively identified on wide strip, it seems probable that it is the cause of slow pickling. One way of preventing the formation of this film, by sealing the edges of the hot coil with boric oxide after removal from the coiler, has been tried out successfully in two mills, but the spraying of boric oxide is unacceptable under production conditions. Other possible ways of bringing about the desired effect are under consideration.

The effectiveness of the lubrication of the rolls and stock in cold rolling influences the size of mill, number of passes required, thinnest gauge produced, and the tonnage rolled per roll dressing. As yet, little is known about the mechanism of lubrication, nor is it known whether or not it can be improved by other than trial-and-error methods. The assessment of a series of eleven experimental oils on the 14 in. laboratory mill has revealed none better than palm oil. At the same time research into the mechanism of lubrication has demonstrated the possibility of reducing friction and wear by grinding the rolls transversely instead of circumferentially as is done at present.

As the speeds of sheet and strip production increase, so the problem of adequate inspection becomes more acute, in many instances the point having been reached at which human visual inspection cannot keep pace with the high line speeds. To overcome this problem and to meet the exacting demands of high speed inspection, B.I.S.R.A. has developed three types of equipment for automatic in-line inspection. They are an ultrasonic lamination detector, a multi-channel photo-electric scanner, and an arrested image T.V. apparatus.

Because of the superior quality of product obtained, the use of continuous annealing for blackplate has been increasing in the tinplate industry in recent years, but the size and cost of existing annealing plant are large on account of the relatively lengthy time cycle at present considered necessary. To establish whether improvement could be achieved by using some alternative annealing cycle, a metallurgical investigation was made of the recrystallisation, soaking and cooling parts of the process. This work has shown that recrystallisation occurs almost instantaneously at a temperature of about 700° C., even with rapid heating-up. It was also found that by quenching from 700° C. to 200°–300° C., coiling at this temperature and over-ageing the strip for 15 minutes in a non-oxidising atmosphere, the same results could be obtained as by slow cooling from 700° C. to about 60° C. Can-making trials on 5 in. wide strip treated in a rapid annealing plant at B.I.S.R.A.'s Swansea laboratory have confirmed that the properties of temper universal for tinplate can be achieved with a processing time of less than ten seconds. The strip is preheated to about 200° C. in a tank of lead-bismuth

eutectic, passing through an electric induction heater, and is cooled in a second bath of liquid metal. The two tanks are connected by ducts and the metal is circulated by an electromagnetic pump to maintain thermal equilibrium. Plans are in hand to build in a member's works a pilot production annealing plant to this design for 39 in. wide material. Progress is also being made in extending rapid annealing to other types of material, such as thicker gauge sheet for drawing and stamping, and the softer qualities for deep drawing.

Good progress has been made on the development of a process for coating strip with aluminium by electro-phoretic deposition of aluminium powder followed by rolling and heat treatment. When produced under optimum conditions, the properties of the new aluminium coated steel (Elphal) are most attractive. The coating is uniform and thickness can be controlled to fine limits over a wide range. There is very little alloy present and the steel can be quite severely deformed without any deterioration of the coating. The porosity is so low that the surface can be anodised. The raw aluminium powder costs 50% more than the ingot aluminium used in hot dipping, but this is offset by the fact that so little alloy is present, and that the coating is much more uniform in the powder-based product.

The process is carried out in three stages with a single pass. When the strip is passed through a bath containing a liquid suspension of the powder, an electric current passing through the bath causes a migration of the powder towards the strip (electrophoresis). The deposit adheres to the strip sufficiently for it to be dried and transported to the second stage, where it is compacted by a light pass through a rolling mill. The final heat treatment process is carried out in coil form by a new resistance heating technique developed for this purpose.

#### **Mechanical and Physical Characteristics**

During the last fifteen years considerable attention has been paid to the phenomenon of brittle fracture of mild steel at low temperatures. B.I.S.R.A. has carried out investigations into the influence of nitrogen, aluminium, silicon, zirconium, titanium and niobium on the transition temperature of a 0.17% carbon, 0.85% manganese steel. As a result of these studies it has been established that the effect of nitrogen, in the absence of any nitride-forming elements is to enlarge the transition temperature range, without affecting the transition temperature. The transition temperature range is reduced, however, when aluminium, zirconium or titanium is added to steels containing nitrogen.

Under certain conditions the transition temperature itself can be lowered. Silicon additions of up to 0.3%, for instance, will result in a progressive lowering of the transition temperature, but it is raised again by any increase in silicon beyond this level. The influence of individual elements depends, to a considerable extent, on the heat treatment of the steel and the processing conditions to which it has been subjected. In steels which have been normalised and/or quenched and tempered, for example, niobium causes a significant reduction of the transition temperature, whereas in rolled steels it has little effect.

The capacity of the fatigue testing laboratory has been supplemented by the installation of a 6 ton Losen-

hausen direct-stress machine, and is being further increased by a 6 ton Haigh-type machine. Tests on a 1½% nickel-chromium-molybdenum steel, heat treated in various ways to produce a tensile strength of 100 tons/sq. in. and a hardness of 500 V.P.N., have shown that the fatigue limit may vary from  $\pm$  28 to  $\pm$  44 tons/sq. in., depending on the heat treatment. Two samples of the steel which had different micro-structures but almost the same static mechanical properties had markedly different fatigue values. Experiments are in hand to discover the causes of this variation. This work is important in confirming that static mechanical properties are not an adequate guide to fatigue strength.

The efficiency of transformers, alternators and other electrical machines is considerably reduced by the energy lost in their magnetic cores, and the amount of energy lost depends on the quality of the core material. Magnetic cores are usually made of silicon iron, an alloy which can be made into strip with excellent magnetic properties. Unfortunately, these magnetic properties may be seriously impaired by impurities in the alloy, which are thought to produce this effect by (1) modifying the recrystallisation texture, and (2) forming inclusions which hinder the wall movement of magnetic domains and so increase the hysteresis loss. Among elements known to be harmful are carbon and nitrogen, and in the absence of quantitative data carbon has always been considered to be the more harmful of the two. An investigation by B.I.S.R.A.'s metallurgy division has shown, however, that in tests where the recrystallisation texture is kept constant, nitrogen is by far the worse offender.

#### Rapid Analytical Techniques

Industrial chemical analytical procedures are capable of extreme accuracy, but most of them are somewhat protracted and of only limited use for the control of liquid steel in the melting furnace, or for the rapid analysis of large numbers of final samples. In recent years, however, there has been a considerable amount of development in the application of physical and physico-chemical techniques of analysis, and these are now gradually replacing the traditional chemical procedures.

A vacuum direct reading emission spectrometer can be used to determine the percentage of carbon, manganese, silicon, sulphur, phosphorus, nickel, chromium, molybdenum and vanadium and the results reported within 10-15 minutes of receiving the sample from the furnace, and in a number of the larger steelworks the control of the melting furnaces is based entirely on instruments of this type. The direct reading instrument is costly and there is still widespread use for certain purposes of the ordinary optical spectrograph where the results are recorded as spectral lines on photographic plates. A calculating board has been devised for use with this technique which simplifies the conversion of line densities to percentages of elements present.

Both the optical spectrograph and the direct reading version are at their most accurate where low percentage contacts are concerned, but where the contents to be determined are extremely low (up to 0.01%) or much higher than the normal range (down to 5%) alternative methods are desirable. One such method is X-ray fluorescence spectrometry in which the specimen, irradiated by high intensity X-rays, emits secondary X-rays



The AEI basic electron probe micro-analyser

of wavelengths characteristic of the elements it contains and with intensity proportional to their content. This radiation is separated into component wavelengths by a diffracting crystal and the intensities measured by a suitable detector. This technique has been shown to be eminently suitable for the precise analysis of low alloy steels, but its most important application will probably be for the analysis of high alloy steel, ferro-alloys and slags. Although the precision of this method is high, inter-element effects are appreciable; a calculator for these effects has been developed and patented.

An AEI basic electron probe micro-analyser has now been installed in the laboratory. This instrument is capable of analysing a spot of the order of 0.5 micron diameter for elements above atomic number 22 (titanium), and experience in its use will be gained by the study of such inhomogeneities in steel as inclusions, banding, distribution of alloying elements among different phases, surface scales, etc. It will then be used for determining the degree of homogeneity of steels used in the division's research programme, for studying processes occurring in heat treatment, and for investigation with creep, fatigue, corrosion, etc.

In spite of these new expensive techniques, the older physico-chemical methods such as absorption spectrometry still have an important role to play. A case in point is the determination of small amounts of aluminium in steel, which is becoming increasingly important in investigations into such problems as grain-size control, deoxidation, the influence of aluminium on nitrogen content, and its effect on the low temperature properties of mild steel. An investigation by the metallurgy division has shown that the aluminium content can be determined in 4-5 hours using Eriochrome Cyanine compared with about 1½ days required by the British Standard volumetric procedure.

### General

In addition to items featuring the work of the divisions housed at Sheffield—or closely related to it—the operational research department mounted a display showing the many and varied fields in which operational research can help the iron and steel industry. Examples given ranged from laying down economic stock levels to planning efficient information systems, and from reducing transport problems to designing the job to fit the man.

Such matters as patents and developments, technical services and information services were covered by the

development and information services display. The steel users section is located at Hoyle Street and deals with general technical inquiries, industrial surveys, compilation of data on En steels, etc.

Finally, visitors were afforded an opportunity of seeing in one of the courtyards an important industrial 'historical monument' restored for permanent preservation. This was the last of the cementation furnaces, as used in steelmaking for decades before the converter and open hearth furnace were introduced. This particular furnace was still in operation in the late nineteen-forties.

## Alcan's Unique Teleprinter Network

In their London headquarters at Aluminium Canada House, 30 Berkeley Square, Aluminium, Ltd., of Canada (Alcan) have installed one of the most comprehensive privately-operated teleprinter systems in the industrial world. The new network connects the U.K., Canada and the United States in a pattern of high-speed communication which will greatly increase the efficiency of Britain's export-import trade in aluminium. It will now be possible, for example, to transmit orders in a matter of minutes direct to the group's smelters in Canada and to plan the schedules of Alcan's fully-owned shipping company closely in keeping with up-to-the-minute demands.

The first messages on the new system were exchanged on 26th June between Mr. Nathanael V. Davis, President of Aluminium, Ltd., visiting Britain from Montreal, and Mr. George Hees, Canadian Minister of Trade and Commerce in Ottawa. Mr. Davis also sent a message of greeting to the twenty-four United Kingdom offices associated with Alcan. All of these will now be inter-

connected with each other, with the control centre in London and, via trans-Atlantic cable, fifty-seven stations in Canada and the United States through the Montreal switching centre. A further extension of the network to embrace Continental Europe is being planned.

All the facilities of the organisation on both sides of the Atlantic are thus brought within a closely-integrated pattern of operational control. This will streamline the group's services to the metal industry and will expedite sales to the United States, the Caribbean, and South America, of Alcan metal fabricated in the United Kingdom. It will also speed-up Alcan's sales in Britain and other European markets. Last year the North American section alone handled 600,000 company messages. The entire system now provides 12,000 miles of direct communication between Alcan plants, offices and customers. It permits instant contact between such widely separated centres as Kitimat, British Columbia, where Alcan have giant smelters and power plants, and Banbury, in Oxfordshire, where the group has extensive research laboratories and important fabricating facilities.

The complex network utilises the facilities of Canadian Overseas Communications, Britain's General Post Office, the Bell Telephone Company, the Trans-Canada telephone system, the Canadian National Telegraphs, and the American Telephone and Telegraph Company. Creed supplied the equipment for the United Kingdom installations. An important feature of the new system is a high degree of automatic operation achieved by the use of punched paper tape in conjunction with special electronic units. This method, together with the "party line" system which interconnects the twenty-four United Kingdom stations, is not yet in general use in Britain. A similar system is used by airlines for interconnection between different companies, but the Alcan network for industrial and commercial communication is the only one of its kind serving the metal industry.



Mr. Nathanael V. Davis (right) examining the tape of his first message on the new Alcan teleprinter system with the Hon. George A. Drew Q.C., High Commissioner for Canada.

FROM 1st July the welding business of the English Electric Co., Ltd., is being co-ordinated in a newly-formed welding equipment division which is being set up at the company's Accrington works. The manager of the division will be Mr. R. H. Boughton, the chief engineer Mr. F. Mullery, and the sales manager Mr. E. H. Ayres.

THE London office of Crofts (Engineers), Ltd., has been moved from 34, Norfolk Street, Strand, to new premises at 36/42, Tanner Street, Bermondsey, London, S.E.1 (Telephone: HOP 7751/4).

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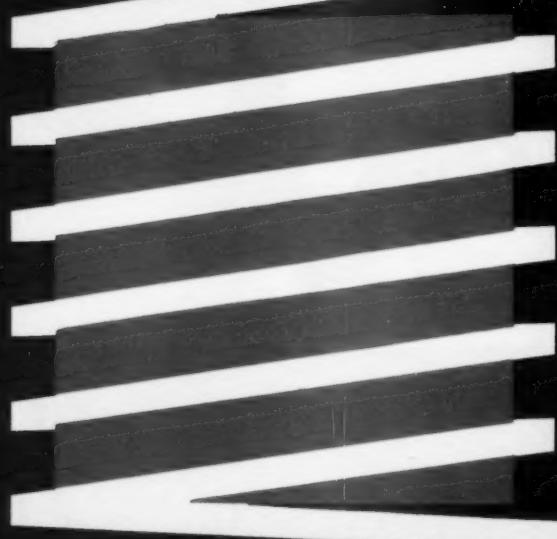
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# NEWS AND ANNOUNCEMENTS

## Beilby Medal and Prize, 1961

THE Administrators of the Sir George Beilby Memorial Fund, representing the Royal Institute of Chemistry, the Society of Chemical Industry and the Institute of Metals, have decided to make awards from the Fund in 1961—each consisting of the newly instituted gold medal with a prize of 100 guineas—to the following:

To CONSTANTIN EDELEANU, in recognition of his work on the corrosion of metals and alloys, with special reference to the development of the potentiostat technique and its applications to the study of practical problems, and on the characteristics of corrosion reactions in fused salts.

To PROFESSOR JACK NUTTING, in recognition of his work in physical metallurgy, especially in the application of the electron microscope to the study of the relationship between microstructure and mechanical properties of metals and alloys and to the investigation of phase changes and dislocation interactions.

In accordance with the revised conditions, the Beilby Medal and Prize will henceforth be offered at intervals of two years, but more than one award may be made on the same occasion, as in this year, if there are several candidates of sufficiently outstanding merit. No further award will therefore be made until 1963.

## £1,000,000 Export Contract for Davy-Ashmore

STROJIMPORT, one of the national buying agencies in Czechoslovakia, has placed a contract with Davy and United Engineering Co., Ltd., of Sheffield, a member of the Davy-Ashmore group, for a highly mechanised plant designed for the production of railway wheels and allied products. This contract, valued at over £1 million, is one of the largest ever placed with the West by Czechoslovakia and also represents the first penetration by Davy-United with heavy plant into socialist countries since the end of World War II.

Designed with a high degree of mechanisation and requiring the minimum of operators, the plant will be capable of sustained high production (initially 200,000 a year) of monobloc wheels, disc wheel centres and tyres for rolling stock. The plant will use the Kendall-Taylor process of wheel manufacture developed by Taylor Bros. & Co., Ltd., of Trafford Park, Manchester, one of the largest wheel manufacturers in the world. The Czechoslovakian plant, based on that originally installed ten years ago at Taylor Bros., follows similar plants built under licence in U.S.A. and Canada. The output will be achieved by numerous complex high-speed handling devices between all the major units of the plant, so that manual handling will be entirely obviated at all stages.

Among the principal units of plant supplied by Davy and United Engineering Co., Ltd., are an ingot block breaking press, furnace charging and discharging machines, a 5,500 ton drawdown type wheel forging press, a wheel mill of patented design capable of rolling at a rate in excess of 65 wheels per hour, and all the specialised inter-unit handling equipment. Other units, such as a rotary hearth block heating furnace, an 800

ton punching press, a 2,000 ton dishing press of special design and plant for the heat treatment of the rolled products prior to machining, will be built in Czechoslovakia to Davy-United drawings. The plant will also include roughing and finishing tyre mills of special design, produced by Wagner & Co., of Dortmund, which, at the special request of the Czechoslovakian buying agency, are also being ordered through Davy-United.

## New B.I.S.R.A. Research Section

B.I.S.R.A. has created a new section in its plant engineering and energy division to centralise the Association's research into control engineering. When the plant engineering and energy division was formed, an electrical engineering section was included to carry out research into the electrical equipment used in steelworks. During the last few years, and with the increasing interest being shown in automation in the iron and steel industry, the research of the section has tended more and more towards the electrical engineering aspects of automatic control systems—their design, construction and application to steelworks plant. Some work on hydraulic control systems has also been carried out in another section of the division. The basic theory of control applies to both, and the one selected depends upon the specific requirements.

In view of the advantages to be gained in bringing all the work in the division on control engineering within the province of one section, the electrical engineering section has been replaced by a new control engineering section. The change of name is a recognition of the gradual change of emphasis in this field of work; the new section will continue the present research into the electrical engineering problems of steelworks with the addition of work on hydraulic control. The Association has appointed Mr. J. P. Clyne, who has recently joined B.I.S.R.A. from the Sperry Gyroscope Co., Ltd., as head of the new section.

## Blast Furnace Refractories

IN the account of the blowing-in of the new blast furnace at the Millom Hematite Ore and Iron Company's works which appeared in our June issue, reference was made to the record run of No. 3 blast furnace which, in an eight-year campaign, made 1,013,474 tons of iron at an average coke rate for the full campaign of 16.23 cwt./ton of iron. For a number of weeks during the last two or three years the furnace was operated on practically 100% self-fluxing sinter and a blast temperature of over 1,000° C., with a coke consumption of under 12 cwt./ton.

The refractories used were supplied by General Refractories, Ltd., the stack being in Foster Crown and Foster High Grade qualities, the bosh in Foster Crown, and the hearth in Foster Carbon. Halfway through the campaign the stack had to be repaired as a result, it was thought, of inadequate cooling. During the repair, more coolers were used in the area of failure and no further trouble was experienced. In view of the experience gained with this campaign a full Foster carbon hearth is to be used again, and increased cooling will be applied to the stack.

## A.D.A. Directory of Members

THE new edition of the Aluminium Development Association's Directory of Members with Index of Products has, in general presentation been brought into line with other A.D.A. publications under the programme of redesign for the entire series. It has also undergone considerable modification and expansion of content. The publication is now divided into two parts, the first being an index of products, whereby a user requiring aluminium in a particular form—castings, sheet, foil, sections, wire, forgings, etc.—is given on sight a list of member companies supplying aluminium in that form, and is referred by page number to the second part of the book in which the full particulars (name, address, sales offices, etc.) of corresponding member firms are set out. Copies of the Directory and Index are available without charge to *bona fide* enquiries.

## H.F. Furnace Order for G.W.B.

G.W.B. FURNACES, LTD., of Dudley, Worcs., have received an order to build what will be the largest and most powerful high frequency induction melting furnace in the United Kingdom. This order has been placed with G.W.B. by the North Eastern Iron Refining Co., Ltd., of Stillington, Stockton-on-Tees. It will be used for the production of highly specialised refined alloy pig irons and steel. The first furnace body will have a capacity of up to 7 tons of molten metal, but the electrical equipment has been so designed that a second furnace body may have a capacity of just over 10 tons. The plant will be housed in the main cupola melting shop to allow for duplexing operations and machine casting.

The Stillington installation, for which the North Eastern Electricity Board is providing a new overhead supply will comprise a 1,700 kW. high frequency generating plant working in conjunction with a bank of water-cooled capacitors. A motorised 10,000 A. furnace selector switch is also to be supplied to enable the high frequency power to be switched to either of two furnaces. The equipment, which will be engineered and built by G.W.B. to an approved design of Demag Elektrometallurgie, is expected to be commissioned in December of this year.

## GKN House

UNTIL 1960 the London headquarters of the GKN group of companies was located at 66 Cannon Street, E.C.4., where there was room for only a limited number of representatives of group companies, other companies having offices in many different parts of London. This somewhat unsatisfactory state of affairs has now been remedied by the erection of a modern office block on the site of the old Stoll Theatre in Kingsway, W.C.2.

Known as GKN House, the new block faces Kingsway on the south-west, and on the north and east overlooks Lincoln's Inn Fields and the Law Courts. It is a seven-storey building with 92,000 sq. ft. of office accommodation, furnished and equipped in the most modern style, and two basements for car parking. In bringing together all existing London offices, the aim has been to serve the group and its customers with maximum efficiency and satisfaction, and to provide amenities and working conditions for the staff of such a standard that as a building it will retain its modernity in years to come.



View of GKN House as seen from Kingsway.

In addition to the office accommodation on the upper floors, GKN House provides on the ground floor showrooms where a selection of products manufactured by the group is displayed. Adjacent to the showrooms is a cinema and lecture theatre accommodating fifty people for the use of group companies and their visitors.

## Nickel Price Increase

To assist in offsetting higher and mounting costs since the company's last price rise four and a half years ago, The International Nickel Co. of Canada, Ltd., has announced new and increased prices for its refined nickel, which will be £660 per long ton in the United Kingdom, representing an increase of £60 per ton. Appropriate increases are being introduced for other countries, and new prices are being announced for other forms of primary nickel.

In announcing the present price changes, International Nickel pointed out that, since the last rise in the nickel price on December 6th, 1956, the company has absorbed mounting costs of all kinds. The company cannot, however, continue to absorb cost increases of the magnitude it has been carrying, which have greatly exceeded the benefits of efficiencies and increases in productivity brought about by the company's process research and more efficient capital installations. As a part of the company's policy of stimulating the expansion of uses for its nickel in preference to other materials, through providing steady prices for its customers over the longest possible periods, it has deferred any change in its prices until this time.

## Nuclear Power Contracts for TI

A £300,000 CONTRACT for electrically-welded finned steel tube, for the Dungeness nuclear power station, has been received by the Tube Investment company, Tube Products, Ltd., of Oldbury, Birmingham. This follows contracts for similar extended surface welded tubing, to a total value of over £2 million, placed with Tube Products for the Hunterston and Trawsfynydd nuclear power stations in the United Kingdom; the Latina project in Italy, and the Tokai-Mura project in Japan.

Tube Products pioneered the development of this type of extended surface tubing some three years ago, and has already supplied over 2 million ft. of the material.

### B.I.S.R.A. Conference

B.I.S.R.A. metallurgy division announces a conference on "Hydrogen in Steel," to be held at Harrogate from 11th to 13th October next. The specific aspects of the problem to be discussed include: (1) removal of hydrogen from liquid and solid steel; (2) the diffusion and solubility of hydrogen in steel; (3) the effect of hydrogen on the properties of ultra-high tensile steels; and (4) hydrogen in weld metal. Full details and application forms will be available from 14th August, when applications should be made to the Technical Secretary, Metallurgy Division, B.I.S.R.A., 11 Park Lane, London, W.1.

### "O for Oxygen"

THIS year the British Oxygen Co., Ltd., celebrates its seventy-fifth anniversary, and to mark the occasion the company has made a film called "O for Oxygen." It shows how Priestley's discovery of oxygen in 1774 has stemmed a world-wide use of oxygen and other industrial gases in industry, medicine, aviation and scientific development. It also illustrates how the various ingredients of the air are being used to make steel more efficiently, to cut ships' plates more quickly and more accurately, to weld metals more strongly, to send up rockets, and to save lives.

Made for general interest by World Wide Pictures, Ltd., "O for Oxygen" is photographed in Eastman Color by Technicolor and runs for 23 minutes. Copies of the film in 16 mm. and 35 mm. will soon be available on free loan from B.O.C. to schools, colleges and similar organisations.

### European Aluminium Consumption Doubled by 1970

SPEAKING at the recent Light Metals Congress at Leoben, Austria, Mr. D. Hamer, president of Alcan S.A., Zürich, European sales distributor for Aluminium, Ltd., of Canada, predicted a doubling of the current aluminium consumption by 1970 in Western Europe. Mr. Hamer estimated that European smelter capacity would be expanded from 870,000 tons to 1,500,000 tons within the decade. Expansion of smelter capacity beyond that would, he said, "be restrained, particularly by the growing realisation that Europe cannot condone the increasing wasteful use of its fuel resources . . . essential to the future."

Examining the trend in the major areas of aluminium consumption, such as the building, transport, household appliances, electrical and packaging industries, Mr. Hamer estimated that European consumption would increase from just below 1½ million tons in 1960 to at least 2½ million tons within the decade. By that time, however, Europe's capacity for smelting aluminium would be about 1,500,000 tons. Its production deficit, therefore, which was 410,000 tons in 1960, would then be at least 800,000 tons. To produce this additional amount in Europe would be extremely difficult in view of the continent's shortage of power resources and ever-

increasing need for electricity. To meet the expected future demand from aluminium consumers, the European fabricating industry would need to be considerably expanded and modernised, and would be obliged to import substantial additional tonnages of primary aluminium from outside Europe.

Mr. Hamer said that supply of primary aluminium for Europe "cannot be satisfied automatically by the probability of a world surplus" and pointed out that "the European market will find it necessary to create import conditions which will not impose penalties on foreign suppliers . . . Tariffs on imported aluminium represent a danger to the European fabricating industry in the long term. Their elimination in favour of effective anti-dumping legislation used by some countries will go a long way to ensuring adequate supplies of metal at all times."

### Personal News

THE appointment has been announced of MR. R. P. MACKAY as superintendent, steel plants, of the steel division of The Steel Co. of Wales, Ltd., with effect from 1st July. Mr. Mackay joined the company in 1934 as a student trainee at the Margam coke ovens.

SIR PETER ROBERTS, B.T., M.P., the RT. HON. LORD RIVERDALE, D.L., J.P., SIR ROBERT ADEANE, O.B.E., J.P., MR. F. H. BROOKS and MR. K. E. WALKER have been appointed to the board of Hadfields, Ltd.

HILGER & WATTS, LTD., announce that MR. A. H. CAMPBELL, who has been a director of the company since 1951 and general manager since 1954, has been appointed joint managing director with MR. G. A. WHIPPLE.

MR. W. H. DAY has been appointed to the board of the Wellman Smith Owen Engineering Corporation, Ltd., as commercial director, and has relinquished his position as secretary, in which he is succeeded by MR. D. G. FELTON. MR. C. D. WATTLEWORTH has been appointed assistant managing director (technical) and MR. D. A. HUME assistant managing director (commercial). MR. A. T. EASTERBROOK has been appointed general manager of the furnace building and contracting department in succession to MR. G. TALBOT, who has resigned from the company's service.

WITH the advent of new processes of steel making and new strip mills the markets available to The Steel Company of Wales are constantly changing. The higher management of the company, appreciating this, considers that it is now appropriate to unify and strengthen its research activities which have previously been organised on a divisional basis. To implement this, DR. C. S. BALL, at present superintendent of research, steel division, has been appointed research controller of the company, responsible to the managing director, with the duty of developing and increasing the scope of the company's research facilities. This will include the co-ordination and, where appropriate, unification of the present organisations. It is intended that a start will be made immediately on construction of the research laboratories at Port Talbot.

MR. R. P. MOORE has been appointed the representative of the process heating division of Pye, Ltd., for the area south of London and the southern counties, and MR. R.

**LAPISH** will cover the East Anglian area, including Stamford, Wellingborough and Stevenage. **MR. J. J. TYRRELL** will continue in the north London area and High Wycombe, with **MR. J. A. TAYLOR** covering South Wales to Birmingham as far as the Yorkshire and Lincolnshire border.

**THE UNITED STEEL COS., LTD.**, announce that **MR. J. D. WRIGHT** has been appointed manager of the Templeborough melting shop of the Steel, Peech and Tozer branch from 1st July. He succeeds **MR. H. H. ENGLAND**, who has resigned.

**MR. H. R. BROOKER** joined the board of Johnson, Matthey & Co., Ltd., on 15th June, 1961, as a joint managing director of the company. Mr. Brooker has hitherto been general sales manager and he will continue to be principally responsible for all the sales divisions of the company.

**MR. C. G. CONWAY** has been appointed sales manager of Great Lakes Carbon International, Ltd.

**MR. R. A. DAY** relinquished his position as chief metallurgist of Blackburn Aircraft, Ltd., at the end of June to take up an appointment as chief metallurgist of Wellworthy, Ltd., Lymington, Hants. **MR. R. BOLER** succeeds Mr. Day as chief metallurgist of Blackburn Aircraft and Blackburn Engines, Ltd.

**MR. F. HOLLOWAY**, secretary of the Workington Iron and Steel Co. branch of The United Steel Cos., Ltd., has been appointed secretary and office manager on the retirement of **MR. W. McGREGOR**. **MR. G. HIRD** has succeeded **MR. J. S. BOOTH** as agent responsible for the company's mining and quarrying properties in Cumberland.

**MR. G. H. JOHNSTON** has been appointed managing director of the Meltham Silica Firebrick Co., Ltd., one of the Genefax group of companies: he has held the position of general manager since 1954. Mr. Johnston joined General Refractories, Ltd., in 1934 and was successively manager of the Wharncliffe works and Lowood Silica Brick Works before moving to Meltham.

**THE GISHOLT MACHINE CO. (GREAT BRITAIN), LTD.**, announce the appointment of **MR. S. W. PERKINS** to the board of directors. Mr. Perkins is a director of Wickman, Ltd., sole agents in the United Kingdom for Gisholt machines.

**MR. G. TALBOT** has taken up an appointment as deputy managing director of Stein & Atkinson, Ltd.

**MR. C. C. HODGSON** has retired from his position as chief metallurgist to Leyland Motors, Ltd., after more than forty years' service. After studying metallurgy at Sheffield University, Mr. Hodgson joined the company in 1918, being appointed assistant chief metallurgist in 1923, and chief metallurgist three years later. He is succeeded as chief metallurgist by **MR. C. S. JOHNSON**, manager of Leyland's Farrington foundry for the last four years. **MR. J. FERGUSON**, the foundry's production manager, has been appointed foundry manager.

**DR. T. P. HOAR** left the United Kingdom on 11th July for a private visit to the United States, after which he will lecture on the corrosion of metals at the University of Chile's Postgraduate Engineering Winter School in Santiago. This visit is sponsored by the British Council and during his stay in Chile, from 19th August to 22nd September, he will make advisory visits to industrial plants and laboratories.

**MR. E. BEALING** has been appointed chairman of Sandvik Steel Band Conveyors, Ltd., in succession to the late **MR. J. F. SJOGREN**.

AFTER forty-eight years' service with The General Electric Co., Ltd., **MR. A. B. PRICE**, sales manager of the company's Erith works, retired at the end of June.

## Obituary

We regret to record the death of the following:

**DR. A. G. C. GWYER**, formerly scientific manager to the British Aluminium Co., Ltd., who died suddenly on 27th June, 1961, whilst on holiday in Cheltenham. Born in 1880 of an old Bristol family, Dr. Gwyer lived for virtually the whole of his working life and retirement in Lymm, Cheshire.

A graduate of London University, he took his doctorate at Göttingen, and also received part of his education at the Universities of Bristol and Birmingham: he was a Capper Pass Metallurgical Scholar and an 1851 Royal Exhibitioner. Commencing work at the National Physical Laboratory in 1911, Dr. Gwyer joined the British Aluminium Co., Ltd., as research metallurgist in 1912, becoming chief metallurgist in 1920, and scientific manager in 1939, from which position he retired in 1945. During his time with the company, there took place under his direction many notable developments in the evolution of aluminium and its alloys, and he was the author and joint author of numerous scientific papers in this field.

Dr. Gwyer was a firm believer in institutional work and was an original member of the Institute of Metals, later serving as member of council and vice-president, and a founder member of the Institution of Metallurgists. He was also president of the Manchester Metallurgical Society shortly after its foundation. In addition he was prominently associated at various times with the work of the Welding Research Council, the Institution of Welding, the British Standards Institution and the British Non-Ferrous Metals Research Association.

**MR. G. SANDLAND**, of The International Nickel Co. (Mond), Ltd., who died suddenly when returning from a company sports outing at Birmingham on 8th July, 1961. Mr. Sandland, who will be remembered by many for his vigorous activities on his plating campaign, had been with the company for thirty-five years.

Trained as an engineer-designer with Vickers-Armstrong, he joined the late Sir William Griffiths and Miss E. W. Parker to form the triumvirate who transformed the development and research department of the Mond Nickel Company into a highly valued asset. He saw the very first issue—and many subsequent issues—of the *Nickel Bulletin* through the press, to a point where it was considered a major contribution to metallurgical literature. During the war he worked with unbounded enthusiasm in the development and research department in the company's London office, and it was there, for the first time, that his health broke down and a long period of treatment and rest was imposed. After the war he rejoined the publicity department, where he once again distinguished himself by his inventive mind and characteristic zeal. Latterly he had been prominent in the nickel plating labelling scheme, which is now reaching a state of maturity.

# FLASH

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and aluminium  
alloy at-  
1500 lb. per hour



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the best of the world's furnace designs

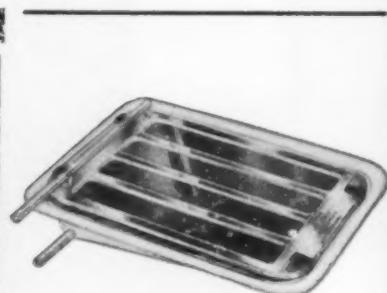
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Replacing the usual improvised grinding plates, this simple inexpensive assembly presents four 11 in. x  $1\frac{1}{2}$  in. silicon carbide waterproof abrasive surfaces continuously washed by water jets. £12 plus £6.6. for four 50 ft. rolls of abrasive paper.

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This equipment drastically reduces man-hour costs of specimen preparation—can polish any number of specimens from 2 to 18 at one loading without attention. £120.

**4 Micro-Hardness Tester**

Applying loads of 1 gram to 200 gram, this is an ideal unit for the hardness testing of all small parts, including 0.0005 in. foil, fine wire, case hardened parts, etc. £280. £340.

*Also available (not illustrated):*

**Electrolytic Polisher.** This removes the surface structure of the metal by electrolysis and is ideal for single and multiphase alloys.

**Electron Microscope.** For metallurgical work—resolution 20 Angstroms, magnification better than 100,000.

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# RECENT DEVELOPMENTS

## MATERIALS : PROCESSES : EQUIPMENT

### Flame Controllers

Ether, Ltd., have developed a new range of flame controllers for safely igniting and monitoring multi-burner installations in industrial concerns. They are fully transistorised, with plug-in circuitry and A.C. coupled amplifier, and are designed for use with gas-fired and oil-fired burners or burners working on a combination of both fuels. Moreover, they discriminate between flame and flame-radiation, and are unaffected by the latter.

Series 704 flame controllers can be supplied for controlling up to six flames at once, depending on customers' needs. Each flame is controlled by a 'flame-eye,' comprising a lead-sulphide cell and a transistor amplifier which is sensitive to the modulated component in the flame-radiation. The presence of the flame is indicated by an output signal from the transistor amplifier which, in turn, switches a silicon transistor. The circuitry is so arranged that the pilot relay, which interlocks with the flame-relay and safe-start relay, cannot be energised until all the transistors are switched to 'flame-on' positions. Absence of one or more flames will de-energise the pilot relay and indicate an alarm condition.

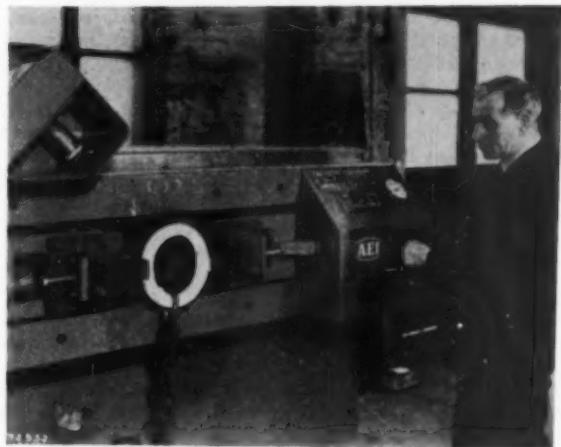
When switched on, the internal main connection is delayed for a pre-purge period that can be varied between 30 and 120 seconds. After the delay an ignition light switches on, then a push-button is pressed which causes a solenoid valve in the pilot-burner line to open. Simultaneously, the ignition transformers produce sparks at the pilot-burner tips which ignite the pilot burners. The 'flame-eyes' then see the flames and immediately register the 'flame-on' position, whereupon the main solenoid valve is energised to open the main fuel supply. If the burners are controlled by a modulating valve, the interlocking contacts (which until now have held the valve in the 'low-fire' position) operate and allow the burners to go on control. If flame-failure occurs, the pilot valve and main valve close, the control valve closes to 'low-fire' position, and the alarm signal is given.

*Ether, Ltd., Combustion Safeguard Division, Caxton Way, Stevenage, Herts.*

### Chain-Link Normaliser

Associated Electrical Industries, Ltd., has introduced a new machine for the normalising or full heat treatment of steel chain links. Developed by the company's heating and welding department in conjunction with the Parsons Chain Co., Ltd., it will apply heat to an untreated link in a chain such as a joiner link, avoiding the costly reheating of the remainder of the chain. Although designed for links of from  $\frac{1}{4}$  in. to  $\frac{1}{2}$  in. diameter, it may be used occasionally to heat links of larger diameter. It is particularly useful in the assembly and repair of steel lifting chains to B.S. 1663, B.S. 2902, etc.

The complete equipment, including transformer and control gear, is mounted in a fabricated steel structure designed to withstand rough handling. Facilities are



**A.E.I. chain-link normaliser in use, showing photo-electric cell, die blocks and control box.**

provided for the addition of a salt bath when full heat treatment is required for high-tensile alloy-steel chains.

The links are clamped pneumatically between electrodes conveying heating current from the transformer, which is provided with a range of tappings to give adjustment of the current. Electronic control can also be provided to give stepless variation of the current over an even wider range, and to enable the current to be altered during the heating of a link. A photo-electric cell cuts off the heating current when a preset temperature is attained, thus preventing over-heating of the chain.

The die blocks that hold the link and also act as electrodes are mounted on sliding carriages. The position of the left-hand block can be preset by means of a screw and nut to suit any length of link; the right-hand one is pneumatically controlled by a hand valve, the pressure applied to the air cylinder being adjustable through a reducing valve.

*A.E.I. Heating & Welding Sales Dept., Trafford Park, Manchester 17.*

### Electronic Air Filter

The Trion electronic air filter, marketed by W. C. Holmes & Co., Ltd., since 1956, has been redesigned to meet the arduous operating conditions encountered in industry, and it will in future be known as the Industriion electrical air filter.

The new filter is of much sturdier construction than its predecessor, and the insulators are now positioned out of the main air stream; this facilitates cleaning and ensures greater reliability. There are now two standard sizes of power units which, in various combinations, meet the power requirements of individual installations, and the electrical equipment includes inherent current limitation which protects the equipment in the event of

a short circuit. The larger unit is fitted with a constant voltage transformer to provide a stable and effective performance under varying atmospheric and dust loading conditions.

A further development is the introduction of a special heavy duty filter which is intended for use on applications where the dust burden is higher than that handled by the standard filter. With this heavy duty filter, pre-collectors of the multi-cell cyclone type are normally fitted to arrest any large particles which may be present in the extract system. Each heavy duty filter installation will be individually designed after careful consideration of the data available, pilot plant tests being carried out on site where necessary. A filter arrangement comprises an ionising section followed by one or more collecting plate sections in series. The combination of these sections is determined by the collection efficiency required and dust holding capacity necessary to give reasonable periods between cleaning.

As opposed to the standard filter which is cleaned by washing, heavy duty filters are normally arranged for dry removal of the dust, the complete filter section being vibrated to dislodge the collected dust which falls into storage hoppers below the filter. The cleaning operation occupies only a few seconds and the filter can be immediately returned to line. One of the advantages of this system of cleaning is that the filter can be frequently cleaned, thus allowing a high dust burden to be handled.

The Industriion range is completed by the Industriion oil mist precipitator, which has been specifically designed for the elimination of oil mist and smoke emanating from high speed cutting, grinding and machining operations.

*W. C. Holmes & Co., Ltd., Turnbridge, Huddersfield.*

### New Way to Trim Lawn Edges

A new and inexpensive means of keeping garden lawn edges straight and tidy without constant cutting back has recently been introduced by The British Aluminium Co., Ltd. It consists of a roll of aluminium strip, 4in. wide, and 33 or 100 ft. long, which goes into the ground round the lawn, and being flexible, fits any shape of border. The top of the strip, which is lightly corrugated for strength, is placed just below the level of the lawn, so the mower blades can pass over it. This is



Aluminium strip being placed in position at edge of lawn.

deep enough to prevent the grass roots spreading on to the path or the beds. The edges of the strip have been turned down, so there is no risk of cuts while fixing it. This consists simply of scraping away the earth from the edge of the lawn, putting the edging alongside just below the level of the grass, and replacing the soil. No supports or pegs are needed, and no hammering is required. Being made of aluminium it cannot rust and has an indefinite life. Like edging is already used extensively in the United States.

*The British Aluminium Co., Ltd., Norfolk House, St. James's Square, London W.1.*

### Starter with Single-Phasing Protection

The motor and control gear division of Associated Electrical Industries, Ltd., has just introduced an entirely new 5 h.p. starter, known as the DOC. 80, for three- and single-phase motors. This is believed to be the lowest priced unit on the market giving single-phasing protection on three-phase supplies. An ambient temperature compensator is incorporated to allow for differences in local temperature between the starter and the motor.

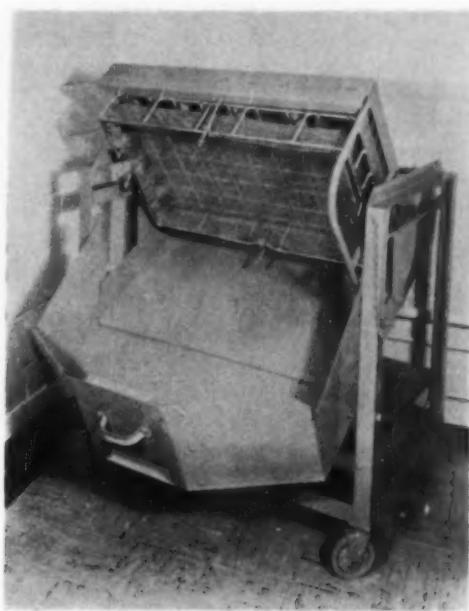
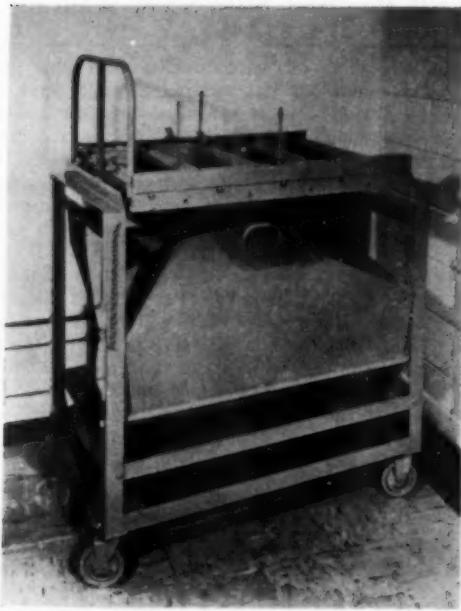
A set of overload heaters is supplied separately in a small pack; the heaters can be fitted very quickly to make the starter ready for service. There are fifteen different heater ratings, which, with the calibration control, enable the relay to give precise protection of the motor. The starters are supplied fitted with any one of five coils covering standard voltages. These, together with the range of fifteen overload heaters, will give seventy-five variations of the starter. Thus, only the minimum stock of starters is required to cover a wide range of ratings—a considerable advantage where customers' and stockists' shelf space and inventory are limited.

The starter can be easily mounted into position, three-point fixing being carried out from inside the case without having to remove any component. Wiring is also simple and leads can be taken in and out of the starter from the top, bottom or side.

*Associated Electrical Industries, Ltd., Motor and Control Gear Division, Industrial Control Gear Sales, Marsden Mill, Nelson, Lancs.*

### Handling of Small Parts During Heat Treatment

A COMBINATION dump cart and hopper, manufactured by Ipsen Industries Inc., is designed to simplify the handling of small parts during heat treatment operations. Basically, the dump cart consists of a dolly with a tilting roller platform and a hopper. In operation, the basket load of parts is rolled onto the platform where the basket is clamped into position. A drain pan under the rollers collects excess oil accumulated during quenching operations. The hopper chute is then opened, and the platform is rotated to pour the parts into the hopper. An opening at the narrow end of the hopper chute permits controlled feeding of the parts into tote boxes or other containers. With the hopper chute folded in, the platform can be locked in the horizontal position, allowing the unit to serve as a dolly. Mounted on wheels, it can be rolled into position for the manual loading or un-



Mounted on swivelled wheels, the Ipsen dump cart (left) serves as a dolly and can easily be rolled into position for loading or unloading heat treating units. In the dump position (right) the work basket is locked in place, permitting the parts to pour into the hopper. The opening at the narrow end of the hopper allows parts to pour into tote boxes under complete control.

loading of a battery of furnaces in any sequence desired. The dump cart permits oil soaked loads to drain and the hopper eliminates spillage and saves handling time in removing treated parts from the work basket.

*Ipsen Industries, Ltd., 53 Victoria Road, Surbiton, Surrey.*

### Logarithmic Electronic Recorder

A BRITISH designed electronic strip chart recorder with alternative logarithmic or linear response, to be made by Honeywell Controls, Ltd., at Newhouse, Lanarkshire, is intended mainly for research applications, though the instrument will find industrial uses.

The recorder has two slidewires fitted in the standard position and the pen carriage is fitted with two sets of slidewire contacts. One slidewire is included in the measuring circuit and, to obey a logarithmic law, it is wound in twelve linear sections, each section having wire of different gauge or material. The law accuracy of the logarithmic slidewire is better than 0.4%, state Honeywell. The second slidewire is of linear construction and this is connected to a D.C. power pack from which a negative bias voltage is applied to the final voltage stage of the amplifier via the second set of slidewire contacts. Thus the magnitude of this bias voltage is relative to the pen carriage position and so provides automatic amplifier gain control to maintain stable operation over the full scale travel.

A two-position switch is incorporated in the measuring circuit to provide logarithmic or linear working. The new logarithmic/linear recorder has a calibration accuracy of better than 1%, but to maintain a repeatable

accuracy of 0.25% over the full scale the minimum span is restricted to 5 mV.

*Honeywell Controls, Ltd., Greenford, Middlesex.*

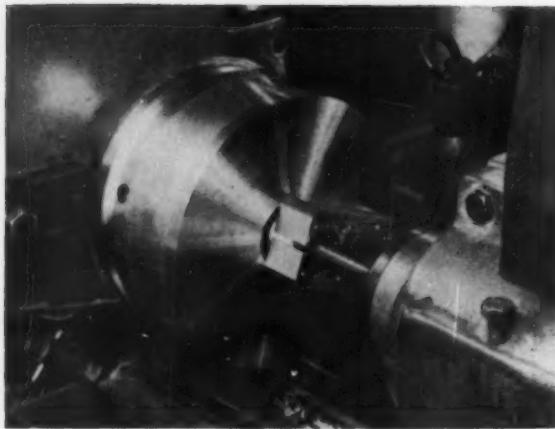
### Absorptiometer-Nephelometer

THE Sigrist photometer UP2/LD is a new combined absorptiometer and nephelometer for laboratory use. It uses the fully automatic optical compensation method unique to Sigrist photometers, whereby all measurements made are a function only of the structure of the light path, being independent of instrumental variations such as light source, photocell sensitivity and amplifier gain. Various versions are available, with or without inkless recorder and motor-driven monochromator. The absorption range with filters is 366-950 m $\mu$ , and with monochromator 400-1,000 m $\mu$ ; the turbidity range is  $2 \times 10^{-5}$  to  $6 \times 10^{-2}$  absolute units.

*Southern Analytical, Ltd., Frimley Road, Camberley, Surrey.*

### Circular Magnetic Chuck

THE Darwins Fimax circular permanent magnet chuck has a high quality manganese-bronze body with radial poles which ensure maximum holding of the workpiece without distortion. A unique feature of the chuck is the removable central adaptor plug which, when in position, ensures that the magnetic flux extends over the full chuck face area. The adaptor is secured by a cap screw which prevents rotation, enabling large or small components to be held firmly for machining. Removal of the screw and adaptor plug enables drilling, boring,



**The Darwins Fimax circular chuck incorporating Alcomax III permanent magnets.**

reaming and grinding operations to be performed at one setting.

Spigoted workpieces can rapidly be set concentric, using non-magnetic bushes, eliminating the need of indicator clocking, while bored workpieces can be set concentric, by use of a stub mandrel, both being useful features for repetition work. Means of chuck mounting may be incorporated to suit specific requirements.

The chucks incorporate Alcomax III permanent magnets, produced by Darwins, Ltd., which are inspection controlled at every stage of manufacture, and which ensure a chuck of extreme holding power. Front to back measurements are as small as possible commensurate with high magnetic flux, enabling the chucks to be run at high speeds since they are normally in good balance. Dynamically balanced chucks are available for very high speeds. Sizes range from 6 in. diameter  $\times$  2½ in. thick to 24 in. diameter  $\times$  3½ in. thick, the normal maximum speed for the former being 3,000 r.p.m. and for the latter 750 r.p.m.

*Darwins, Ltd., Magnetic Tool Division, Tinsley, Sheffield, 9.*

### Proportional Controller

FOR stepless proportional control of electrical loads using magnetic amplifier and saturable reactor, a new millivoltmeter indicating instrument is announced by Honeywell Controls, Ltd. The Pyr-O-Volt controller 105R12 is an inexpensive current-proportioning instrument with no contactors. It has a continuous output of 3-7 mA. working into an impedance of 4,000 ohms or more when used as the input to a magnetic amplifier. It will control saturable core reactors up to 100 kVA., with provision for control of larger reactors. Among important features are the straight line control, automatic fail-safe, built-in voltage regulation, plug-in design, adjustments on the instrument itself, connection and servicing from the front. In the same family is another new instrument, the millivoltmeter indicator 105X11, which has a high quality moving coil movement and a plug-in design.

*Honeywell Controls, Ltd., Greenford, Middlesex.*

### All-Position Mild Steel Welding Electrode

PHILIPS announce the introduction of a new general purpose electrode for the welding of mild steel which will provide weld deposits of regular appearance in all positions—downhand, vertical, overhead and in inclined planes. This electrode, known as type 28, is the latest addition to the Philips range and assists the welder in producing quality welds which are virtually self-deslagging and of excellent appearance. Moreover, it is claimed that it will cope easily with all types of weld joints—fillet, butt, lap and external corners, etc., in all positions which arise in fabrication of mild steel in all thicknesses, having remarkable tolerance of bad fit-up. A special feature of this electrode is that the stability of the arc is such that in addition to first class control over the weld pool in position welding, touch welding techniques may be adopted by the operator. The type 28 electrode complies with B.S. 639 (1952) and is supplied in four gauges—12, 10, 8 and 6 s.w.g. It may be used on either A.C. or D.C.

*Research & Control Instruments, Ltd., Instrument House, 207 King's Cross Road, London W.C.1.*

### Forced Cooling for Vacuum Furnaces

GAS cooling with a fan for forced circulation is now incorporated in automatic vacuum heat treating units manufactured by Ipsen Industries, Inc., a feature that improves metallurgical results and lowers production costs. To operate the unit, the charge is placed in the furnace and the door is shut and sealed. The operator only has to set temperature and time to start the unit through its automatic cycle. After pumping to a pre-set vacuum, the work is heated. At the end of the soak cycle, an inert gas is introduced into the chamber and a centrifugal fan forces the gas along the finned cold wall and then up through the work load. The atmosphere path is controlled to direct it through the work at optimum velocity and with minimum turbulence. When the work reaches 300° F. (150° C.) an alarm sounds to inform the operator that the work can be unloaded.

*Ipsen Industries, Ltd., 53 Victoria Road, Surbiton, Surrey*



**Ipsen force-cooled vacuum heat treatment furnace.**

# CURRENT LITERATURE

## Book Notice

### OSZILLOGRAPHISCHE POLAROGRAPHIE MIT WECHSELSTROM

By J. Heyrovský und R. Kalvoda. pp viii + 198, Akademie-Verlag, Berlin 1960, Price DM. 23.

OSCILLOGRAPHIC polarography by means of alternating current has been developed almost entirely by Heyrovský and his co-workers in Czechoslovakia. Numerous papers have been published but, apart from a small volume by Heyrovský and Forejt in 1953, this is the first book on the subject. It provides an authoritative survey on the theory and basic principles and collects together most of the important published information. The various direct and derivative methods of working are described and the effects of parameters such as temperature, frequency, and capacity of the electrode system are considered. The effect of the reversibility of the system on the oscillogram obtained is described and kinetic phenomena are also discussed.

A description of the electronic apparatus is given and some circuits are described in detail. This section is shorter than is desirable in view of the novelty of the method. Details of the commercially-available instruments, known as "Polaroscopes" are presented.

The most widely applied oscillopolarographic method is a derivative technique in which  $dE/dt$  is plotted against the potential,  $E$ . This gives rise to an elliptical trace on the cathode ray tube screen. The oxidation, or reduction, of depolarisers as a result of the applied potential cause changes in the slope of the voltage-time curve at constant current, and these appear as "cuts-in" on the elliptical trace. The position of the "cut-in" on the voltage axis is characteristic of the species and its depth is a measure of concentration. The shape of the "cut-in" and the general dynamic nature of the whole trace makes accurate quantitative determinations difficult, so that the method is best suited for qualitative studies. Semi-quantitative, or even quantitative determinations are often possible, however, and one of the most valuable applications of the method is in the study of reversibility of metal complex species and other systems. Chapters are devoted to descriptions of methods for measuring the "cuts-in," including a number of instrumental devices. These include the use of a double beam cathode ray tube with a calibrated Y-shift on the reference beam and methods of oscillographic and comparative titration.

Important chapters describe the oscillopolarographic behaviour of many metallic and other inorganic species, many organic substances, and such materials as poisonous gases and industrial vapours. This information is valuable because the most important application of the technique at present is the qualitative, and sometimes semi-quantitative characterisation of species, the oscillopolarographic behaviour of which is often quite different from that observed by classical polarography. Many species which do not yield a conventional polarographic step may be examined by this method.

The final chapter deals with newer instrumental devices designed to produce a standing wave, instead of

the more usual dynamic wave, on the cathode ray tube screen. One of these is the use of a synchronised dropping mercury electrode. Such developments should lead to an improvement in the quantitative results obtainable. A bibliography of 172 references is appended.

This book must be welcomed as an important addition to polarographic literature. It must be criticised, however, on the grounds that it tends to make the technique appear more important and universally applicable than is the case at present. It is well written and adequately illustrated and a good balance between the various topics has been maintained. It should be read by all polarographers. It is however, in German and its wider appeal to more general analysts, although desirable, will probably be limited until the production of an English translation.

## Trade Publications

CAMBRIDGE INSTRUMENT COMPANY's List 143/1 describes their recording counters which record the frequency, clock time, and duration of industrial processes or machine operations. The circular charts on which this information is traced also indicate, by default, periods when the machine or process is shut off. A very wide range is manufactured to allow the best possible choice for any one application. Single- or multi-pen recorders, operated by either mains or battery, are available and combined counters and temperature or pressure recorders can be supplied.

WE have received from The L. S. Starrett Co., Ltd., a catalogue giving details of the hand and power hacksaw blades produced by the company's modern factory at Jedburgh in Scotland. This publication also contains a guide to hacksaw blade selection for cutting various types of material and a chart designed to indicate the causes of trouble in power hacksaw operation.

"RARE GASES, SCIENCE, INDUSTRY," is the title of a profusely illustrated publication issued by Oxyton, Ltd., which features a history of the rare gases whose discovery eventually led to the formation of a new, large scale industry. The use of these gases in illuminating engineering gives rise to a number of striking and colourful illustrations, whilst a section on the efficiency of an inert environment deals with their use in a wide range of industrial processes.

Two types of hump-back conveyor furnace are described in leaflet RF.7 recently sent to us by Royce Electric Furnaces, Ltd. These furnaces are particularly suitable for bright heat treatment and brazing where a protective atmosphere of hydrogen or cracked-ammonia is used, and where it has to be maintained in a high state of purity. The heating elements are of heavy gauge nickel-chromium wire for temperatures up to 1,000° C. (W type) and heavy grade nickel-chromium tape for temperatures up to 1,150° C. (T type). The leaflet contains the full specification and ratings and sizes of the standard units, which range from 10 to 36 kW.

DRAINS to take strong chemical effluents present a difficult problem to the civil engineer. In the construction of a mile-long 48 in. diameter drain at L.C.I.'s Wilton Works, glazed stoneware pipes were used jointed with an Araldite epoxy resin. The technique used is described in *Technical Notes No. 220*, issued by CIBA (A.R.L.) Ltd., Duxford, Cambridge.

A NEW leaflet on stud welding attachments is announced by Crompton Parkinson, Ltd. This publication illustrates a wide selection of both standard studs and studs of special shapes. The choice of studs is virtually unlimited and Crompton Parkinson can make welding attachments of almost any shape and size to the customer's own specification. The leaflet also gives a list of Crompton Parkinson (Stud Welding) branches, overseas licensees and concessionaires.

'HOT DIP GALVANIZING FOR BUILDERS' describes briefly the properties of hot galvanized coatings. Its main function however is to inform architects and builders about the suitability of hot galvanizing as a modern protective finish for a very wide range of fixtures and fittings for buildings. The building industry is seeking constantly to improve quality, and the durability of hot galvanized windows, with which architects and builders will be familiar, should convince them of the merits of this low cost finish to protect all other iron and steel articles from rust. Copies may be obtained from the Hot Dip Galvanizers Association, 34 Berkeley Square, London, W.1.

PRIMARILY intended as a comprehensive brochure to describe the new version of the company's argon chromatograph, a new publication from W. G. Pye & Co., Ltd., contains details of many techniques which have recently been developed in the field of chromatographic analysis. Reading through its pages one realises that, in three short years, the modern gas-liquid chromatograph has established itself as a powerful, and indispensable, tool in the hands of research workers in every branch of chemical science; from medicine to the production of heavy organic chemicals; from perfumery to the detection of impurities in industrial gases.

THE Crusilite heating elements made and marketed by Morganite Electro-Heat, Ltd., are of one piece tubular section, having a spiral as the hot zone. This avoids the necessity for joints between the hot and cold zones and, together with a silicon carbide element considerably improved in respect of ageing, makes for longer element life. A new brochure describes these elements and illustrates their application in laboratory and industrial high temperature furnaces. The simplest elements have a central spiral with electrical connections at the two ends, but another type is novel in that the electrical connections are both at the same end and is intended for applications when it is difficult or impracticable to accommodate the conventional element. Copies of the brochure may be obtained from the company at Point Pleasant, Wandsworth, London S.W.18.

IMPALCO Tube-in-Strip, featured in a recent leaflet from the Imperial Aluminium Co., Ltd., is a single piece of aluminium providing a combination of parallel tubes and strip material. During casting of the rolling slab, rods of a special inert material are introduced and when the slab is rolled, these rods become elongated in the direction of rolling. The resultant strip contains parallel narrow

slits filled with the inert material and, by applying hydraulic or pneumatic pressure to these slits, they can be inflated to form tubular passageways of circular or special shape as required. Tube-in-strip, which is supplied either coiled or in straight lengths, lends itself to heat transfer applications between fluids of differing heat transfer characteristics, e.g. liquid and gas. Other uses include the production of radiant heating panels by inserting heating elements in the tubular passageways.

Since they are highly resistant to many chemicals and to abrasion, Araldite epoxy resins form an excellent basis for screeds for flooring. A formulation is now available for use with sand, which gives a hard floor with exceptional resistance to a wide range of chemicals, particularly inorganic acids and alkalis. An attractive feature of the floors is that they can often be laid with the removal of none or only a little of the existing floor. Full particulars are given in an illustrated article which forms *CIBA Technical Notes No. 222*, obtainable from CIBA (A.R.L.) Ltd., Duxford, Cambridge.

A RAPIDLY growing segment of the aluminium fabricating and manufacturing industry is that devoted to the production of rigid foil containers, which have found widespread application in the preparation and marketing of food. The June 1961 issue of *Aluminium News* features an article on this development illustrating the wide variety of containers which can be made. A somewhat more technical article deals with the stitching of aluminium sheet, which is a well established and versatile process applicable to many industries. *Aluminium News* is published by Alcan International, Ltd.

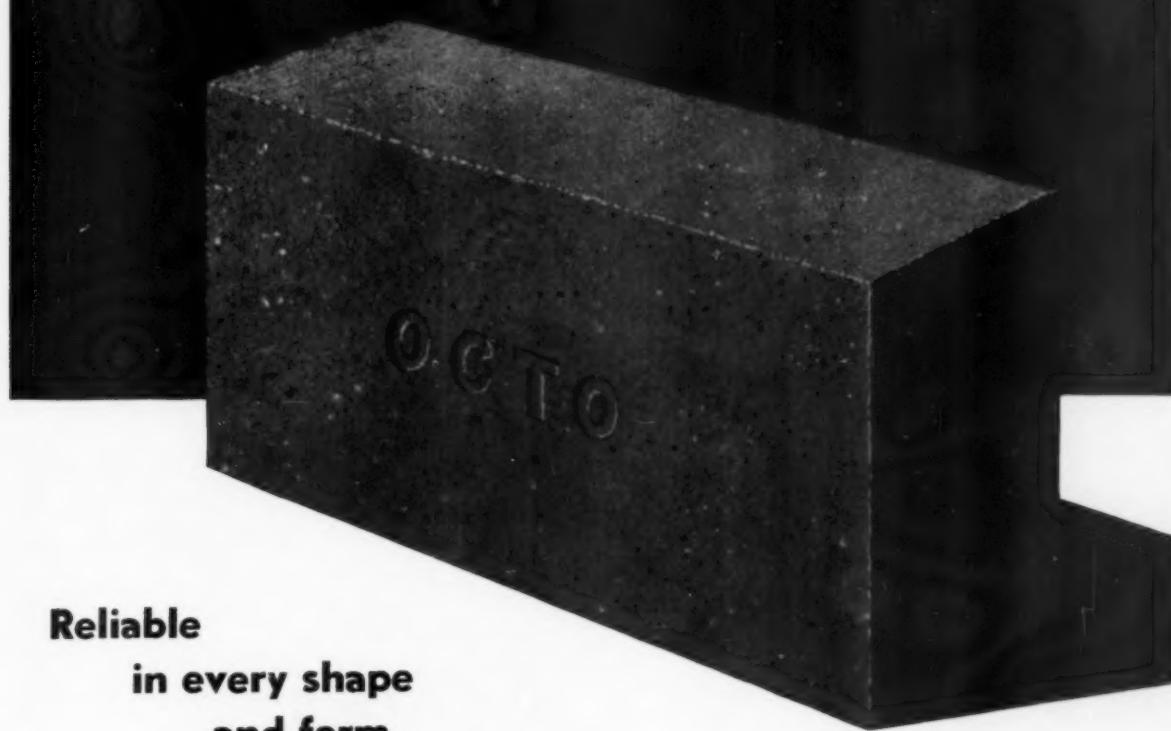
WE have received from the gas cleaning division of W. C. Holmes & Co., Ltd., of Turnbridge, Huddersfield, a brochure (publication No. 87—Holmes-Elex Electrical Precipitators), which describes the principles of electrical precipitation and also practical application of these principles to the many dust control problems encountered in a wide variety of industries. In the iron and steel industry, such units find application in the blast furnace plant, descaling plant, desiliconisation plant, L.D. converters, electric arc and induction furnaces, and open hearth furnaces, and are particularly valuable where fumes from oxygen lancing are involved. In the aluminium industry, electrostatic precipitation is used in bauxite calcining kilns and for cleaning the waste gases from aluminium reduction furnaces. It can also be used for detarring various types of manufactured gas and for dust and mist removal in sulphuric acid plants.

"IRON CASTINGS FOR INDUSTRY" is the title of an illustrated booklet prepared with the assistance of the British Cast Iron Research Association, and issued by the National and Midland Ironfounders' Association, 69 Harborne Road, Edgbaston, Birmingham 15. It is felt by the Association that in recent times, there has been a tendency to use newer and more expensive materials when, in fact, cast iron would have fulfilled all the requirements necessary at a lower cost. The booklet aims at assisting buyers, designers, draughtsmen and others concerned in the specification of materials, to assess more accurately the merits of iron castings. Following a section dealing with the structure and mechanical properties of cast iron, there are others dealing with corrosion resistance, machinability, accuracy, long life and wear resistance, and a table summarising data on grey cast iron.

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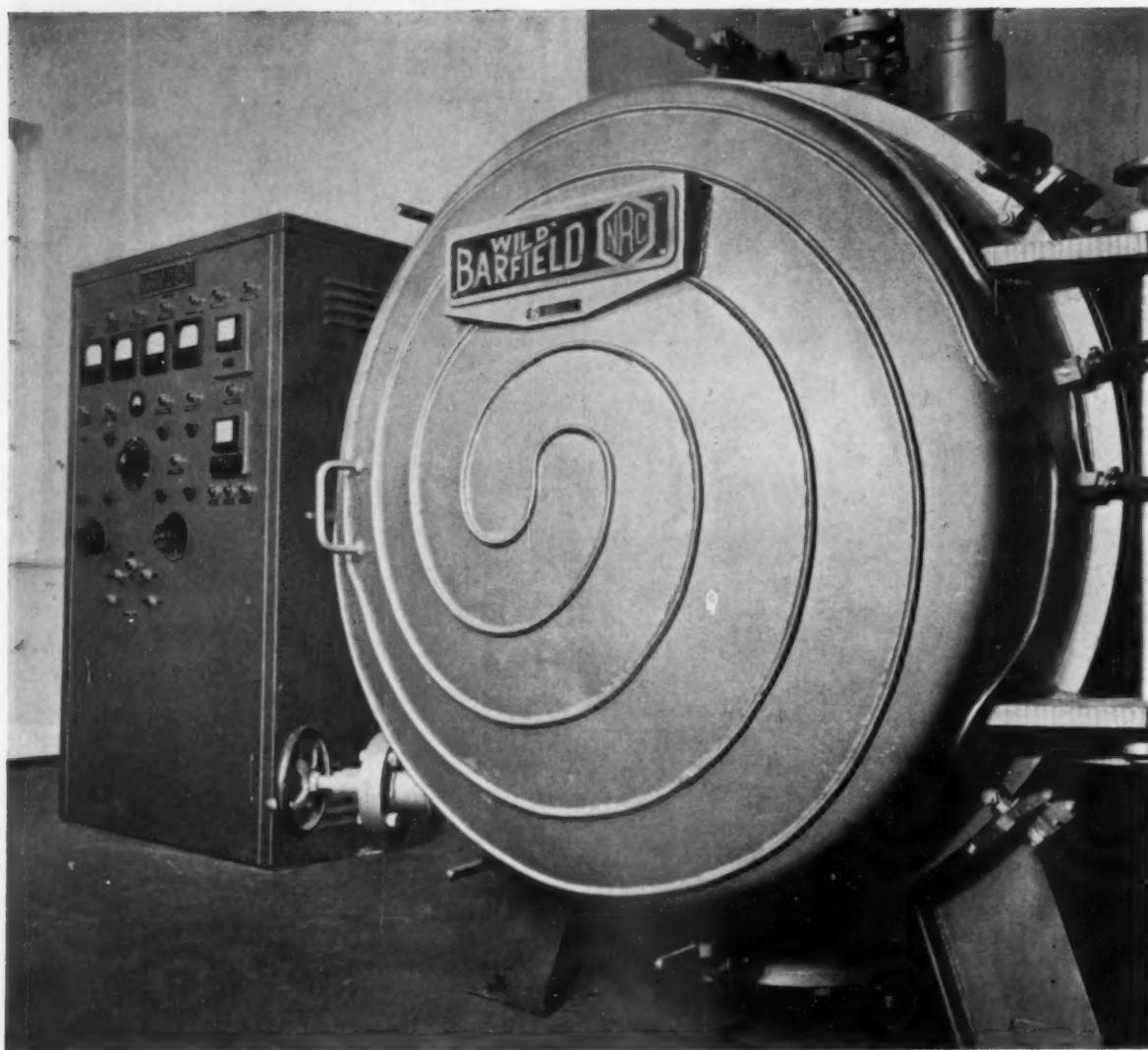
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# LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC  
INSTRUMENTS AND MATERIALS

JULY, 1961

Vol. LXIV, No. 381

## The Spectrophotometric Determination of Small Quantities of Cobalt in Plain and Alloyed Steels

By A. E. Sherwood

Works Chemical Laboratory, The English Electric Co., Ltd., Bradford

A new method has been developed whereby cobalt contents down to 0.005% in plain and alloyed steels may be determined spectrophotometrically as the cobaltous chloride complex in strong hydrochloric acid after the removal of interfering elements by anion exchange and amyl acetate extraction.

IT became necessary to determine cobalt contents down to 0.005% in carbon and alloy steels in the B.S. 970 series by a simpler and more rapid routine method than those in current use. A review of present methods, including that of B.I.S.R.A.,<sup>1</sup> showed that these are complicated and suitable for use only by skilled personnel. The procedure described herein requires only the simplest materials and can be completed in 24 hours by comparatively unskilled workers, the greater part of the time requiring no attention from the analyst.

Previous work in this laboratory has shown that cobalt may be determined spectrophotometrically as the complex chloro-anion in strong hydrochloric acid.<sup>2</sup> Such solutions show absorption maxima at 625, 660 and 685 m $\mu$  (Fig. 3) and as little as 5 mg./ml. may be measured accurately. It was decided to try to adapt this technique for the determination of cobalt in steels.

The biggest obstacle to overcome was the separation of small amounts of cobalt from interfering elements. The method of Hague, Maczkowske and Bright<sup>3</sup> using anion exchange to remove cobalt and iron as chloro complexes was thought the best possibility, this to be followed by the amyl acetate extraction of any residual iron.

### Experimental

#### Absorption Spectra

The work of Lindley<sup>2</sup> established that a strong stable colour is obtained with solutions of cobalt in 387-430 g. HCl/litre. For this work the solvent acid was standardised at 414 g. HCl/litre (S.G. = 1.175), which may be obtained by the slight dilution of concentrated hydrochloric acid. In this way error is minimised, although in this range of acidity the effect of variation in acid concentration is very small. It can be seen in Fig. 1 that a variation of  $\pm 10$  g. HCl/litre results in a maximum error in optical density reading of  $\pm 0.01$  units.

#### Interference from Other Elements

A consideration of standard specifications for these types of steels showed that elements other than iron and cobalt likely to be present are: carbon, sulphur, silicon, tungsten, niobium, selenium, zirconium, tantalum, aluminium, nickel, titanium, vanadium, lead, tin, copper,

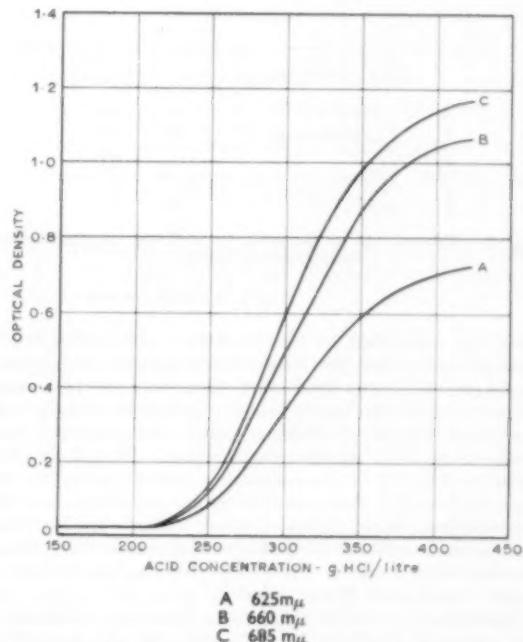


Fig. 1.—Effect of acid concentration with 1 cm. cells and 0.12 mg./ml. of cobalt.

molybdenum, chromium, manganese, cerium and phosphorus. It was decided that the first nine of these elements would either be insolubilised or volatilised by solution in aqua regia and evaporation to dryness, or would have colourless, non-interfering ions. Accordingly, investigations were concentrated on determining the possible interference from iron and the remaining eleven elements.

Standard solutions of these elements, iron and cobalt in 1.175 S.G. hydrochloric acid were prepared using the metals as such where available, or a compound which

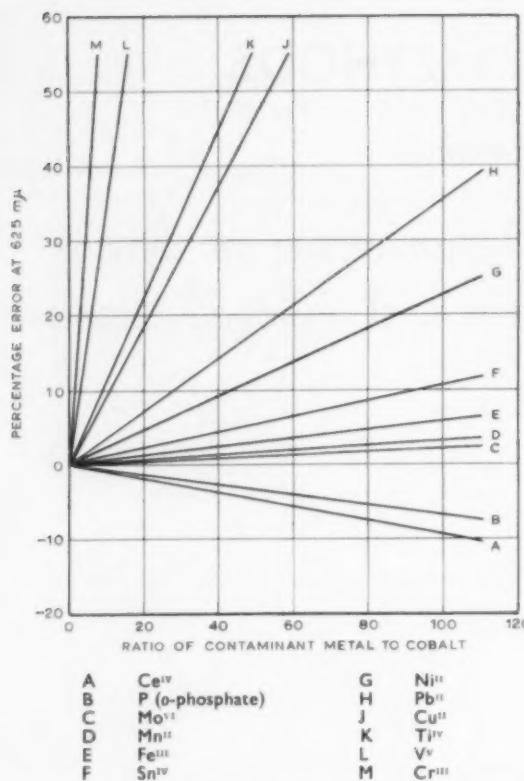


Fig. 2.—Percentage error due to various contaminant-metal cobalt ratios.

could be converted to the chloride. Orthophosphoric acid was used in the case of phosphorus and where necessary a nitric acid oxidation was carried out. Aliquots of interfering metal solutions were added to a measured volume of standard cobalt solution and then made up to a standard volume with 1.175 S.G. hydrochloric acid. The optical densities of the mixtures were measured at 625, 660 and 685 mμ on a Unicam S.P.600 spectrophotometer using 4 cm. cells, 1.175 S.G. hydrochloric acid compensating. Fig. 2 shows the errors incurred as a result of various interfering-element/cobalt ratios based on 0.2 mg. cobalt.

Consideration of the maximum quantities of interfering elements likely to be met, and also the quantities which would lead to a 5% error in the cobalt determination, led to the conclusion that the main effort should be directed towards separating cobalt from iron, nickel, chromium, vanadium, titanium and copper.

#### Separation of Elements other than Iron from Cobalt

According to Hague, Maczkowske and Bright,<sup>3</sup> elution of a mixed metal solution of a high temperature alloy from a strongly basic anion exchange resin column (Dowex 1) with 9N hydrochloric acid, will remove all metals except cobalt and iron, which are absorbed. In their technique, molybdenum and copper are removed beforehand by sulphide precipitation. It was thought unlikely that molybdenum would be present as a serious interference and that copper could be corrected

for if it were so. It is also stated that 4N hydrochloric acid will remove cobalt from the column and leave iron.

Standard solutions of nickel, chromium, vanadium, titanium and cobalt in 9N hydrochloric acid were prepared. These were placed separately upon a column of De Acidite FF ion exchange resin previously prepared in 9N hydrochloric acid, and were eluted with acid of this strength. Nickel, chromium and cobalt eluates were taken to dryness, redissolved in 1.175 S.G. hydrochloric acid, and optical densities measured at respective peak wavelengths. Vanadium and titanium eluates were treated with sulphuric acid and hydrogen peroxide and optical densities of the complexes thus formed were also measured. Blank measurements were carried out on solutions which had not passed through the column. It was found that nickel, chromium, titanium and vanadium passed through the column, chromium the least rapidly, but cobalt was retained in 9N acid and subsequently washed out by 4N acid.

A synthetic alloy mixture containing 0.01% cobalt (0.1 mg.) was dissolved in 9N hydrochloric acid and eluted through the column with acid of this strength, followed by elution with 4N hydrochloric acid. Iron was found in large quantities in the eluate as well as cobalt, indicating that the column was probably overloaded with iron.

#### Amyl Acetate Extraction of Iron

Since the amount of iron eluted with the cobalt was sufficient to give serious error, the amyl acetate extraction technique of Wells and Hunter<sup>4</sup> was used to remove it from the 4N acid eluate, and after evaporation and re-solution in concentrated hydrochloric acid, the optical density reading indicated a cobalt recovery of 95–100%. Even after extraction there was still a small quantity of iron remaining, but insufficient to give a serious error.

#### Analysis of Synthetic and Standard Alloys

Using the recommended procedure laid down at the end of this paper, various synthetic and standard alloys were analysed for cobalt content: the results are shown in Table I.

#### Safeguards and Corrections

As a result of experience gained in carrying out these and other analyses, it was found that the final measured solution is invariably green due to the presence of an unavoidable small quantity of yellow ferric chloride. Since chromium chloride is green, with an absorption peak at 650 mμ, and the presence of only as much of this element as there is cobalt leads to a 5% error, it was

TABLE I

| Alloy   | Cobalt Present<br>%                                | Cobalt Found<br>%                                  |
|---|--|--|
| Synthetic alloys containing Fe, Mn, Ni, Cr, V, Ti, Co | 0.022<br>0.028<br>0.053<br>0.013<br>0.008<br>0.053 | 0.022<br>0.026<br>0.050<br>0.012<br>0.006<br>0.051 |
| R.C.S.220 (W-Mo high speed steel)                     | 0.67   | 0.72   |
| R.C.S.220 + added cobalt                              | 0.87   | 0.91   |
| R.C.S.260/I ("Pure" iron)                             | 0.004  | 0.005  |
| R.C.S.260/I + added cobalt                            | 0.024  | 0.019  |
| R.C.S.219/I (Ni-Cr-Mo steel)                          | <i>x</i>   | 0.028  |
| R.C.S.219/I + added cobalt                            | <i>x</i> + 0.02                                    | 0.05   |
| R.C.S.225/I (Ni-Cr-Mo steel)                          | 0.015  | 0.017  |
| R.C.S.225/I + added cobalt                            | 0.035  | 0.036  |
| R.C.S.235 TIW 18/8 Stainless Steel                    | <i>y</i>   | 0.052  |

Note: *x* and *y* are unspecified.

thought necessary to ensure without any doubt that its presence in the final measured liquid should be detectable and correctable. Accordingly, a series of mixtures of cobalt and chromium chlorides in 1.175 S.G. hydrochloric acid were prepared and examined, their spectra being plotted over the range 600–700 m $\mu$ . Specimen spectra are shown in Fig. 3: it can be seen that there is considerable distortion of the cobalt spectra when chromium is present, the optical densities at the 660 and 685 m $\mu$  peaks tending to equalise. The ratios of the optical densities at these two peaks are shown for a number of cobalt levels, with and without chromium, in Table II. If there is serious chromium interference, the ratio falls below 1.08.

From Fig. 1 it can be seen that optical density at the three cobalt chloride peaks falls to zero in approximately 50% hydrochloric acid. This is not true of chromium chloride, nor, in all probability, in the case of the chlorides of other elements, although these were not studied.

The solution mixtures in Table II were each diluted to 50% and read again at 625 m $\mu$ . A correction was then applied to the chromium-contaminated reading of optical density according to the following equation:

$$\text{Corrected density} = \frac{\text{Density in 1.175 S.G. hydrochloric acid} - \text{twice the density in 50% hydrochloric acid}}{1.08}$$

Cobalt contents associated with the corrected optical densities are now much more accurate. Errors as high as 70% due to 10:1 chromium : cobalt are eliminated. Results are shown in Table III.

### Recommended Method

#### Reagents

Concentrated hydrochloric acid

Concentrated nitric acid

9N hydrochloric acid

4N hydrochloric acid

Hydrochloric acid S.G. 1.175

Amyl acetate

De acidite FF (chloride form) ion exchange resin

#### Special Apparatus

Unicam S.P.600 spectrophotometer with cells etc.

TABLE II

| Cobalt Present (mg./ml.) | Chromium Present (mg./ml.) | Apparent Cobalt (mg./ml.) | $d_{685}/d_{660}$ |
|--------------------------|----------------------------|---------------------------|-------------------|
| 0.02                     | 0                          | 0.02                      | 1.09              |
| 0.02                     | 0.02                       | 0.023                     | 1.06              |
| 0.02                     | 0.04                       | 0.025                     | 1.05              |
| 0.02                     | 0.1                        | 0.028                     | 1.04              |
| 0.02                     | 0.2                        | 0.036                     | 1.01              |
| 0.04                     | 0                          | 0.04                      | 1.085             |
| 0.04                     | 0.04                       | 0.046                     | 1.07              |
| 0.04                     | 0.08                       | 0.050                     | 1.05              |
| 0.04                     | 0.2                        | 0.056                     | 1.03              |
| 0.04                     | 0.4                        | 0.069                     | 1.02              |
| 0.06                     | 0                          | 0.06                      | 1.08              |
| 0.06                     | 0.06                       | 0.069                     | 1.08              |
| 0.06                     | 0.12                       | 0.07                      | 1.05              |
| 0.06                     | 0.3                        | 0.08                      | 1.025             |
| 0.06                     | 0.6                        | 0.097                     | 0.995             |
| 0.08                     | 0                          | 0.08                      | 1.09              |
| 0.10                     | 0                          | 0.10                      | 1.08              |

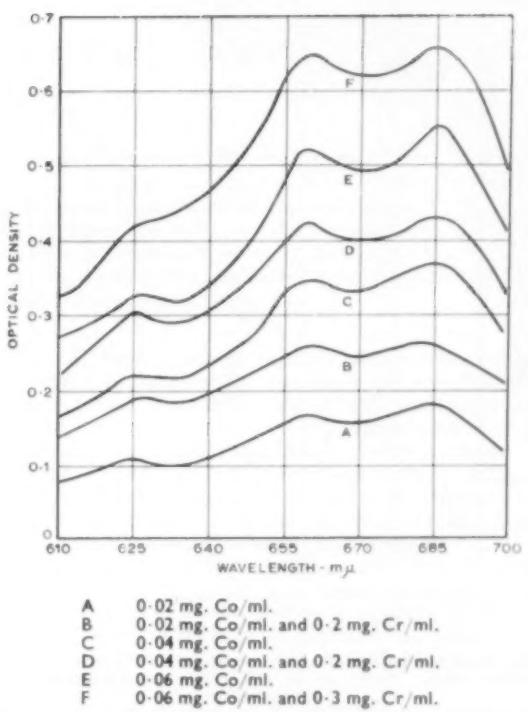


Fig. 3.—Spectra of cobalt and chromium chlorides in 1.175 S.G. hydrochloric acid.

Ion exchange columns to take a resin bed 12 in. long by  $\frac{1}{4}$  in. diameter: the type of column and reagent feeder in use is shown in Fig. 5.

#### Calibration

Prepare a standard solution of cobaltous chloride by dissolving 0.1 g. Matthey Cobalt Sponge JM 870 in the minimum amount of concentrated hydrochloric acid and dilute to 1,000 ml. in a graduated flask, using distilled or deionised water: this solution contains 100  $\mu$ g. Co/ml. Take aliquots of this solution, say 1, 3, 5, 7, 9 ml., pipetted accurately into 50 ml. beakers. Carefully evaporate to dryness and redissolve the residues in exactly 5 ml. hydrochloric acid S.G. 1.175 and read on the spectrophotometer at 625 m $\mu$  in 1 cm. glass cells using acid of the same strength as compensator. Plot optical density against micrograms cobalt or percentage

TABLE III

| Cobalt Present (mg./ml.) | Chromium Present (mg./ml.) | Corrected Cobalt (mg./ml.) |
|--------------------------|----------------------------|----------------------------|
| 0.02                     | 0                          | 0.02                       |
| 0.02                     | 0.02                       | 0.019                      |
| 0.02                     | 0.04                       | 0.019                      |
| 0.02                     | 0.10                       | 0.019                      |
| 0.02                     | 0.20                       | 0.021                      |
| 0.04                     | 0                          | 0.04                       |
| 0.04                     | 0.04                       | 0.041                      |
| 0.04                     | 0.08                       | 0.041                      |
| 0.04                     | 0.20                       | 0.038                      |
| 0.04                     | 0.40                       | 0.039                      |
| 0.06                     | 0                          | 0.06                       |
| 0.06                     | 0.06                       | 0.061                      |
| 0.06                     | 0.12                       | 0.058                      |
| 0.06                     | 0.30                       | 0.057                      |
| 0.06                     | 0.60                       | 0.057                      |

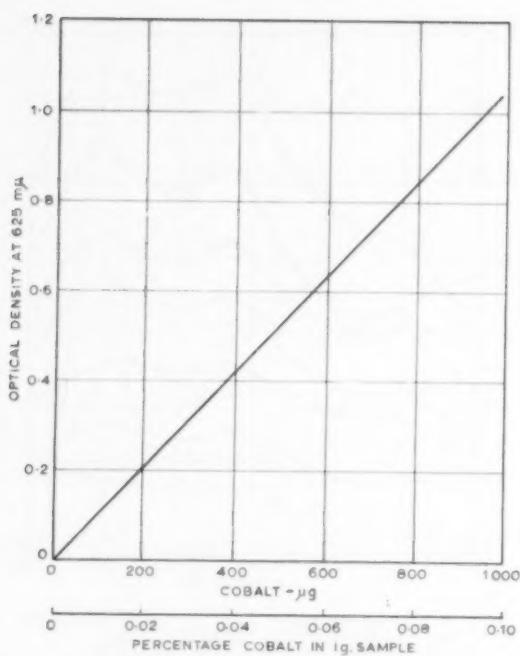


Fig. 4.—Calibration curve—cobalt in 1-175 S.G. hydrochloric acid; 1 cm. cells, 1-175 S.G. hydrochloric acid compensation.

cobalt on a 1 g. sample basis. Such a graph is shown in Fig. 4.

#### Procedure

Weigh a quantity of finely divided sample into a 250 ml. beaker. For steels with cobalt content up to 0.1% use 1 g. sample. If considerably more cobalt is expected use 0.1 g. sample. Dissolve in the minimum quantity of aqua regia and take almost to dryness, first with nitric acid to ensure oxidation of iron, and then once or twice with hydrochloric acid to destroy nitrates. Take up in 50 ml. 9N hydrochloric acid, warming gently if necessary, but do not heat too much or the acid strength will be changed. Material remaining undissolved will not be cobalt, but the solution should not be filtered as the ion exchange column will do this, and at the same time the insoluble material will be washed free of absorbed cobalt.

Prepare the ion exchange column by stirring a suitable quantity of De acidite FF (chloride form) resin with deionised or distilled water. Allow the resin to settle and decant the supernatant liquid. Repeat until this is clear. Place a glass ball or glass wool in the ion exchange tube where it narrows and pour in the resin and water slurry, allowing the water to run to waste until a 12 in. column of resin has been built up. Do not allow the water to fall below the resin level. Run about 200 ml. 9N hydrochloric acid to waste through the column. Pour the sample solution onto the column, allowing it to run through at the rate of about 3 ml./min. Wash the beaker into the column a few times with small quantities of 9N hydrochloric acid, allowing each addition to fall to the resin level before making another. When further additions of acid remain colourless at the top of the column, arrange a gravity feeder containing more 9N hydrochloric acid to produce a total flow of about

600 ml. of acid through the column (see Fig. 5). Allow an acid flow rate of 3 ml./min. If the column is to be left it is advisable to take precautions against dry-up of the resin, as shown in Fig. 5. Reject the 9N hydrochloric acid eluate and then wash the column through with 350 ml. 4N hydrochloric acid from a gravity feeder, as before, and collect it in a 600 ml. beaker (elution rate again 3 ml./min.).

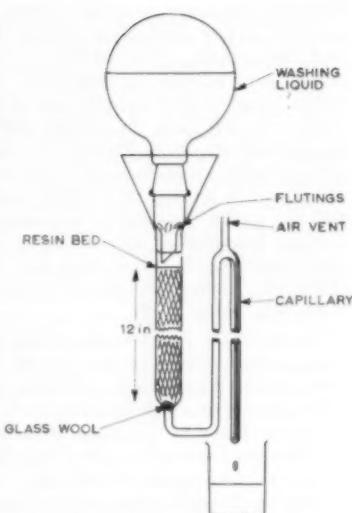


Fig. 5.—Automatic feeder and dry-up prevention device.

Evaporate the 4N eluate to dryness, transferring it to a 100 ml. beaker when the bulk is sufficiently low. Allow to cool. Re-dissolve by adding 5 ml. cold concentrated hydrochloric acid. Transfer the acid solution to a 50 ml. separating funnel. Wash the beaker into the funnel four or five times using a total of 20 ml. amyl acetate. Stopper and shake vigorously for 2-3 minutes. Allow to separate and run off the lower acid layer into a clean 50 ml. beaker and reserve it. Wash the original beaker with 1 ml. concentrated hydrochloric acid and transfer to the funnel. Shake again for one minute and allow to separate. Run off the acid layer into the reserved solution and evaporate to dryness very carefully to destroy dissolved amyl acetate. Re-dissolve the cooled residue in 5 ml. of accurately pipetted hydrochloric acid S.G. 1-175. Read the optical density of this solution at 625, 660 and 685 mμ on the spectrophotometer, using 1 cm. cells with 1-175 S.G. hydrochloric acid compensating. Calculate the ratio  $d_{685}/d_{625}$ . If this is less than 1.08 there may be interfering elements present, and it is advisable to dilute the measured liquid and compensator to 10 ml. with distilled water. Read the optical density at 625 mμ and calculate :

$d_{625}(\text{true}) = d_{625}(\text{in strong acid}) - 2 \times d_{625}(\text{in weak acid})$ . Using the true absorption read off the percentage cobalt in the sample from the calibration graph.

#### Acknowledgment

The author wishes to express his thanks to The English Electric Co., Ltd., for permission to publish this paper.

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ASSOCIATED ELECTRICAL INDUSTRIES, LTD., has received an order from West Germany for a Metroflux Type T universal magnetic crack detector, to be supplied to Klackner Humboldt Deutz, of Cologne, for testing, on a production basis, gas turbine aero engine discs up to 42 in. diameter, with or without blading, and shafts up to 6 in. diameter and 84 in. long.

# New Creep and Stress-to-Rupture Testing Machine\*

By H. Robson

Metallurgist-in-Charge, Creep Testing Laboratory, D. Napier & Son Ltd., London.

*To meet the need for large scale creep testing to keep pace with an accelerated production programme, engineers at D. Napier & Son, Ltd., designed and built, in only five weeks, a machine for testing six specimens simultaneously. An important feature of the equipment is the central tubular column, which serves as a strain carrying member, a furnace support, and a lever fulcrum carrying frame.*

WHEN D. Napier & Son, Ltd., recently embarked on an accelerated production schedule for Eland and Gazelle gas turbine engines, they found that some batches of turbine disc forgings were not fulfilling the stringent requirements laid down for creep resistance. Since the integrity of these units is absolutely vital, it was obviously essential to introduce 100% creep testing of these parts, until such time as experience indicated that a consistent standard could be maintained. The only alternative would have been a severe curtailment of production until replacement forgings could have been obtained.

The result was that the whole capacity of the Napier creep testing research laboratory had to be employed,

and still further testing capacity was needed. Manufacturers of creep testing machines and their ancillary equipment could not offer delivery sufficiently soon, and it became necessary to consider manufacture of the equipment at Napier's own Acton (London) works.

Discussions with the millwright's department indicated that suitable facilities were available to fabricate and assemble such machines on site, provided that no complex forms or shapes were included in the design.

Simplicity of fabrication and assembly from available materials were of prime importance, as also were the restrictions imposed by available laboratory floor space and the high capital cost of the ancillary temperature control instrumentation. The following requirements emerged :

- (1) Only quickly and simply manufactured components to be used.
- (2) The final testing battery to occupy as little floor space as possible.
- (3) Materials to be immediately available (this restriction enforcing certain dimensional limits for components).
- (4) Temperature control and strain reading apparatus to be quickly available and to be kept to a minimum cost commensurate with satisfactory performance.

Consideration of these factors led the author to revive an idea which had been in abeyance for some years as to the possibility of having a radial type of machine in which all tests would be contained within a single furnace. The advantages of this would be that with a single furnace only one controller would be required instead of one for each test point as in the more conventional multi-point equipment. A considerable experience of furnace design gave confidence that the necessary conditions for temperature distribution and accuracy could be achieved in the proposed unit, and the next step was to formulate the mechanical and physical configuration.

## Multi-Purpose Central Column

The study of various methods of construction emphasized the advantages to be gained in limitation of overall size if a single central column could be made to perform the functions of a strain-carrying member, a furnace support and a lever fulcrum carrying frame. This necessitated operating the column at furnace temperature, but its mass within the furnace would assist temperature distribution. As some 7 in. diameter DTD.306 bar

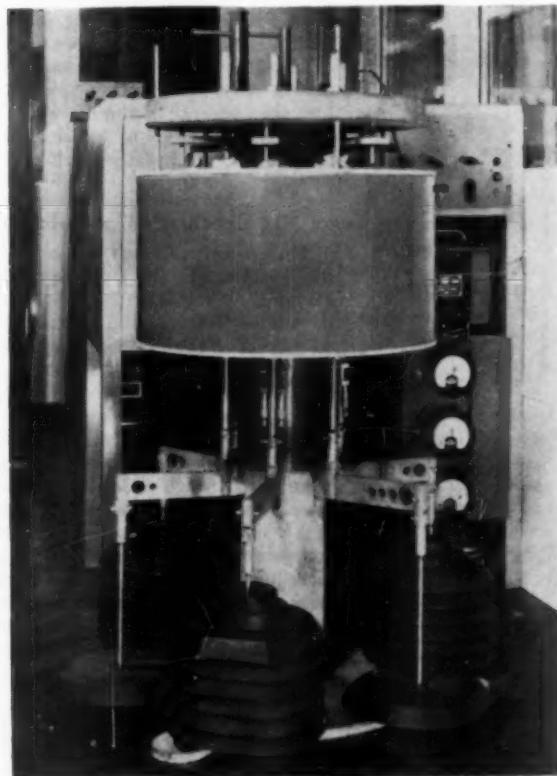


Fig. 1.—General view of the Napier six-specimen creep testing unit.

\* This article first appeared in the January 1961 issue of *Metal Progress*, journal of the American Society for Metals.

stock was available and the maximum required temperature envisaged would be only  $600^{\circ}\text{C}$ . it seemed that the scheme was practical. Taking into account the necessity for the unit to operate under conditions of asymmetric loading, it appeared that a tubular column would be better than solid bar in resistance to bending and this led to the decision to use a tube of  $6\frac{1}{2}$  in. O.D. with 1 in. thick walls, placed into a state of compression by the use of a centrally disposed and axial 1 in. diameter tensile loaded bar, thus forming the most suitable construction possible to resist bending.

The intention to use levers to apply the stresses to the test pieces now demanded a decision whether to use over-slung or under-slung levers, and since the latter would allow the use of the full diameter of the machine in providing insulation for the furnace, the underslung levers were preferred. Consultation with the millwrights regarding the situation of the lever fulcrum knife edges resulted in designing the column in two sections, divided where it would be best suited to these datum points, and made possible the provision of suitable slots in the lower section which would take the ends of the levers and would also allow for the insertion into the

upper section of downwards facing knife edges. These knife edges could be of cyanide hardened mild steel for the male parts, and the levers could have locally hardened "vees" for the female parts.

A rigid circular top plate unit to sit on the top of the vertical column, with holes suitably located to line-up with the lever fulcrums, would allow for the introduction of screw jacks coupled to adaptors to provide means of mounting the test pieces, which in turn could be coupled to the levers. The decision to use levers of a ratio of 5 : 1 would allow enough height from floor level to accommodate a sufficient number of cast iron weights, which were already available, to supply the required range of stresses. Suitable micrometer extensometers of our own design, capable of reading strain to  $0.0033\%$  on a gauge length of 1.50 in. were already available, and electronic transducers capable of greater accuracy were in course of development in the laboratory and should soon be available for use in place of the micrometers.

By this time it was possible to prepare a diagrammatic sketch (Fig. 2) of the projected machine and such was the urgency of the situation that the millwrights began the fabrication of some of the parts which could be fitted

irrespective of the final dimensions of the central column. Meanwhile dimensioned drawings were prepared for a six-point unit. The two halves of the central column and the positioning of the top plate were located by dowel pegs in order to determine accurate alignment of the straining jacks with their respective levers.

All steel parts were cadmium-plated after fabrication, except for the central column, which was nickel-plated to withstand the higher temperature without scaling.

#### Temperature Control

The creep testing laboratory staff fabricated the furnace, the elements being arranged as three parallel windings with separate terminals at their top ends and a common terminal at the bottom. This arrangement allowed the use of a three phase and neutral supply, with each element on a separate phase. Two elements were fed through slidewire resistances and the third through a 1 kW. saturable reactor. The temperature control system used was that the two slide-wires would pass enough power to raise the temperature to about  $100^{\circ}\text{C}$ . below test temperature and the saturable

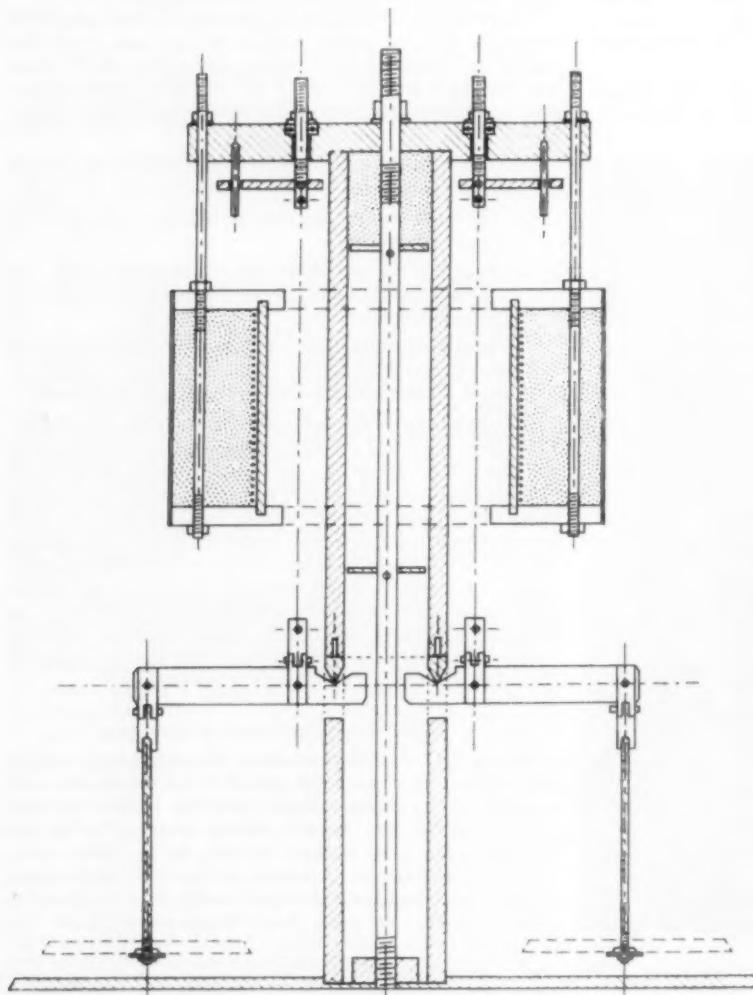


Fig. 2.—Diagrammatic sketch showing the design features of the unit. The main member is a two-piece tube held in compression by a central tiebar so that it does not bend when exposed to test temperatures up to  $600^{\circ}\text{C}$ .

reactor would build up and control the required balance of power input. This system, in which the saturable reactor is controlled by an electronic relay, which is itself actuated by a platinum resistance thermometer situated in the furnace, providing smoothly proportional non-switching control, has been in use in these laboratories for more than seven years, and has proved to be the most reliable and efficient means of temperature control yet experienced.

Final assembly of the completed machine was accomplished within five weeks of the decision to fabricate it, and two weeks were then spent on calibrating the stressing levers and on proving the temperature gradients and distribution. Three thermocouples were attached to each test piece and a single sheathed thermocouple was placed so that its hot junction was positioned midway between two test points and level with the midpoint of the test pieces. The maximum error over all nineteen thermocouples at 550°C. was found to be less than 1°C., and temperature control over 300 hours was to less than  $\pm 0.2$  °C.

#### The Machine in Service

Creep tests of 2,000 hours have since been carried out for comparison purposes, the test pieces all having been taken from a common forging, and the total "scatter"

of results over the six points when tested at similar stresses was less than was experienced on six single point machines, whilst the mean result was identical with a test carried out on a high accuracy machine with a test piece of 4.00 in. gauge length.

At the time of writing, this machine has completed over 10,000 hours of creep testing without interruption of any kind, with complete satisfaction and, moreover, it has been found possible to take off and replace any test without shutting down and without any detectable effect on adjacent tests.

An improved model has since been fabricated and delivered in three weeks to a satellite factory of the company, and it has since performed some hundreds of creep tests in a perfectly satisfactory manner. Patents cover the machine and various adaptations, such as, testing in environmental atmospheres, under cyclic stressing conditions, and under combined creep and fatigue.

#### Acknowledgment

The designer wishes to express his gratitude to Mr. J. K. Wilson, chief metallurgist, and the directors of D. Napier & Son, Ltd., for permission to publish this paper, and to the millwrights for their co-operation in the manufacture of these machines.

## X-Ray Fluorescent Analysis of Metals

### London Applications Laboratory Established by Solartron

THE basic principles of analysis by means of the X-ray spectrum have been well known since Moseley's time (before 1914) but the method remained of academic interest until after 1945, when it attracted attention on a wider scale, due in no small measure to the availability of low-energy counters and small high-power X-ray tubes, and the development of the necessary electronic techniques for complex counting and scaling operations. This renewed interest has resulted in the technique passing from the sphere of the research laboratory to the realms of industrial analysis.

#### Basic Principles

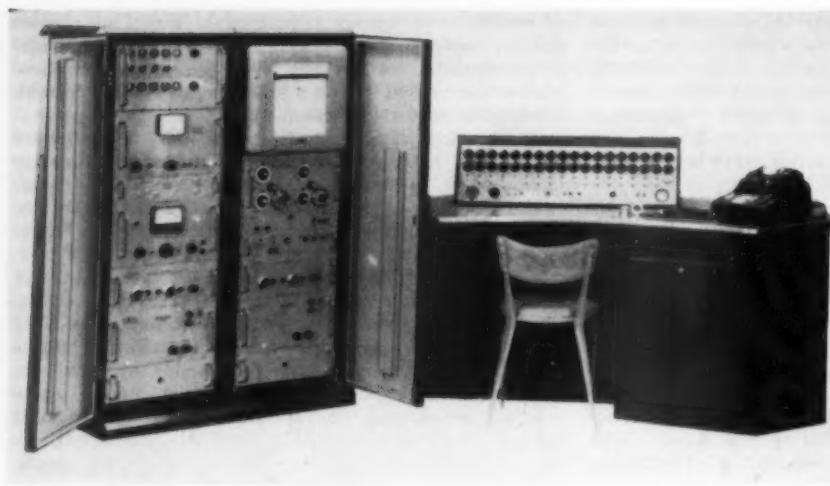
The method depends on the fact that when a beam of X-rays in the 50 kV. region is directed on to a sample, the latter fluoresces and the resulting radiation, which consists of softer X-rays, is composed of the characteristic radiations from each element present in the sample. As X-ray fluorescent radiation is produced by electrons deep down near the nucleus it is independent of any chemical or valency effects, which are characteristic of the outer electron shells. It follows, therefore, that the technique can be used to analyse matter in any form of chemical compound, but it is particularly suitable for alloys.

The intensity of the characteristic X-radiation from a particular element—say copper in brass—is proportional to the percentage of the element in the sample. The separating of the various energies of fluorescent radiation is performed by a spectrometer. The beam of X-rays is collimated by blades of thin sheet held parallel, which thus define the direction of the beam. The beam then

strikes a large flat crystal which performs an analogous function to the diffraction grating in an optical spectrometer. Each characteristic energy of X-radiation has a particular angle (the Bragg angle) at which the crystal reflects the particular radiation strongly, whereas at other angles the radiation is scattered. Since the detector and crystal are rotated (maintaining the correct angles so that any reflected radiation will impinge on the detector), the detector will record peaks of intensity at known angular positions for each element present in the sample. For quantitative detection, a scintillation counter, proportional counter or Geiger tube is used, the fundamental advantage of a counting method lying in the fact that digital techniques can be used throughout and the resulting analyses can be printed-out directly by an electric typewriter.

#### The Solartron Instrument

To meet the need for a fully automatic commercial instrument, The Solartron Electronic Group, Ltd., introduced the XZ 1030 automatic X-ray fluorescent spectrometer after eight years of intensive research and development. This instrument is claimed to be unique in that two samples are irradiated at the same time, one the unknown and the other a reference. In this way, X-ray tube variations, temperature effects and similar drift phenomena are obviated. Thus the unit needs a minimum of calibration or standardising and, although it is advisable to run a check sample through at the beginning of each shift, no adjustment will normally be required. The unknown is compared directly with the reference sample of the same alloy, so that only small



General view of the Solartron XZ1030, showing the electronic rack on the left. On top of the console on the right can be seen the routine operation controls and the pointer. The X-ray generator and main regulator are not shown.

departures are measured. This leads to linear calibration tables and increased accuracy.

In using the instrument for continuous scanning a graphical record is produced showing qualitatively and quantitatively which elements are present. Alternatively, the equipment can be arranged to give a continuous analysis of a given element and to provide a correction or alarm signal if the amount varies outside specified limits. The most common requirement, however, is for accurate quantitative analysis, and for this purpose the programmed counting method is used. The spectrometer is programmed to take up a series of positions—up to twenty-four in number—at which the peaks of the various elements concerned are known to be located. These angles are set up on a series of dials and can be simply altered to suit the analysis. The spectrometer locates each angle in turn, to an accuracy of  $\pm \frac{1}{2}$  minute of arc, measures the number of counts, and prints-out the result opposite the appropriate channel number. This programming is completely flexible, channels being set up and switched in and out of the programme at will. The appropriate programme for diverse selection of completely different analyses can be fed into the spectrometer by a simple punched card.

#### Scope of the Instrument

Specimens should have a surface area of  $\frac{1}{2}$ –1 sq. in. and no lengthy surface preparation is necessary. The speed of operation depends largely on the accuracy required, the longer the time on a given channel the greater the accuracy. In the analysis of a particular alloy the percentage accuracy of minor element contents may not be required to the same high degree as that of major constituents, and the time for the former can be made shorter in setting the programme, thus reducing the overall time. 0.3% of a 50% content element and 0.01% of a 1% content are quite common and usually take less than 10 seconds; repositioning the crystal and the counters takes less than 5 seconds.

The Type XZ 1030 is capable of detecting and determining all elements down to and including atomic number 12 (magnesium) and is particularly valuable in the concentration range from 0.1% to 100%, in contrast to the optical spectrometer, which deals best with

contents from 0.0005% to 5% of most elements in the periodic table. Compositions above 10% cannot generally be tackled by optical methods, whereas it is these regions that are most favoured by X-ray spectrometry.

Copper-base alloys provide an ideal example of the instruments capabilities, and a complex brass containing copper, zinc, lead, nickel, manganese, iron and aluminium can be analysed in a matter of a few minutes with accuracies exceeding routine chemical work. Again, high alloy steels can be rapidly analysed with an accuracy on nickel and chromium contents surpassing other methods.

#### Applications Laboratory

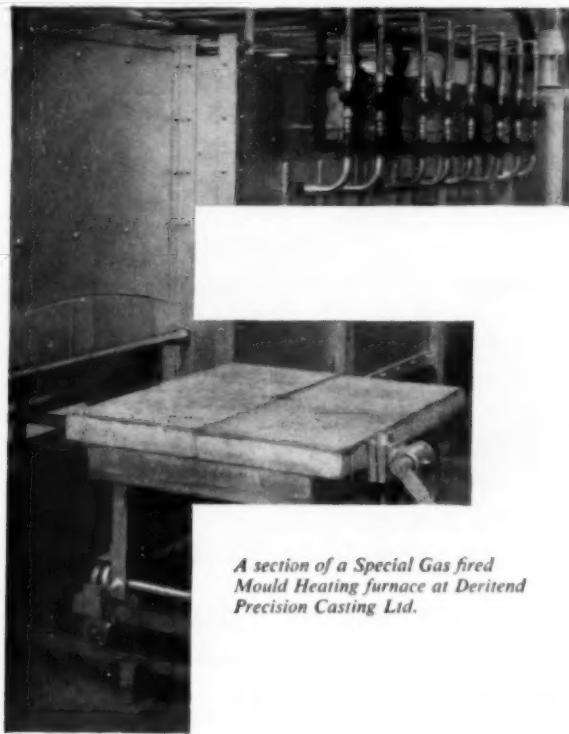
Solartron have set up at the London headquarters of the Firth-Cleveland group—Stormont House, Cleveland Row, St. James's, London, S.W.1—an applications laboratory aimed at providing a threefold service to industry which will be useful to potential customers as well as existing users.

In the first place the laboratory facilitates the demonstration of the instrument to potential users and enables a full explanation of its capabilities to be given.

The second part of the service concerns short-term applications work on customers' samples, thus examining the potential of the instrument performing the exact task required by the customer.

Finally, the laboratory's work is concerned with long-term applications which will be of more general interest. This involves the development of special techniques such as sample preparation, solution work, rapid methods where time is at a premium, etc. Each problem is worked out in detail and the customer is supplied with a set of working curves. A demonstration run-through on his own premises may also be arranged.

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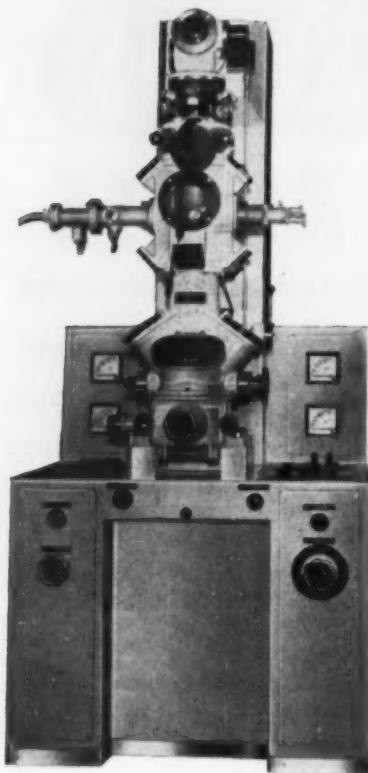
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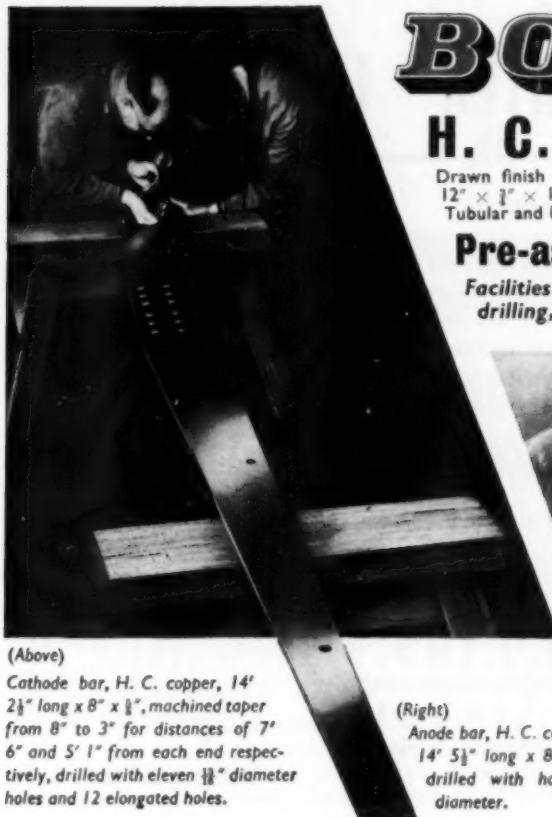
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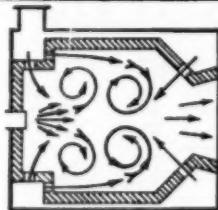
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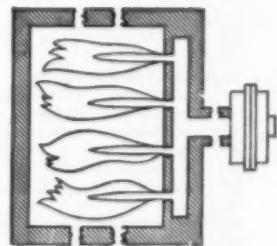
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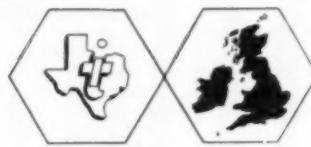
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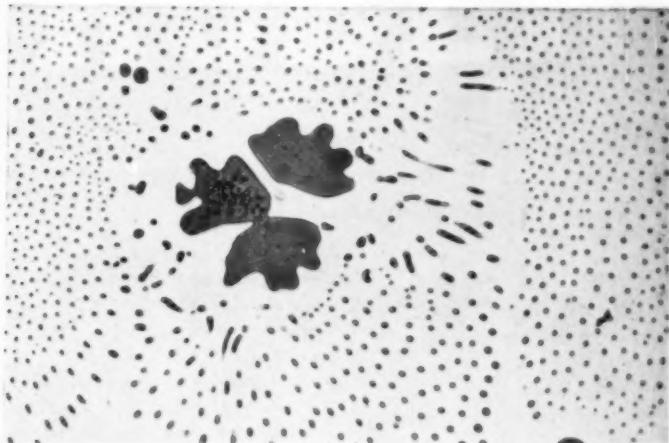
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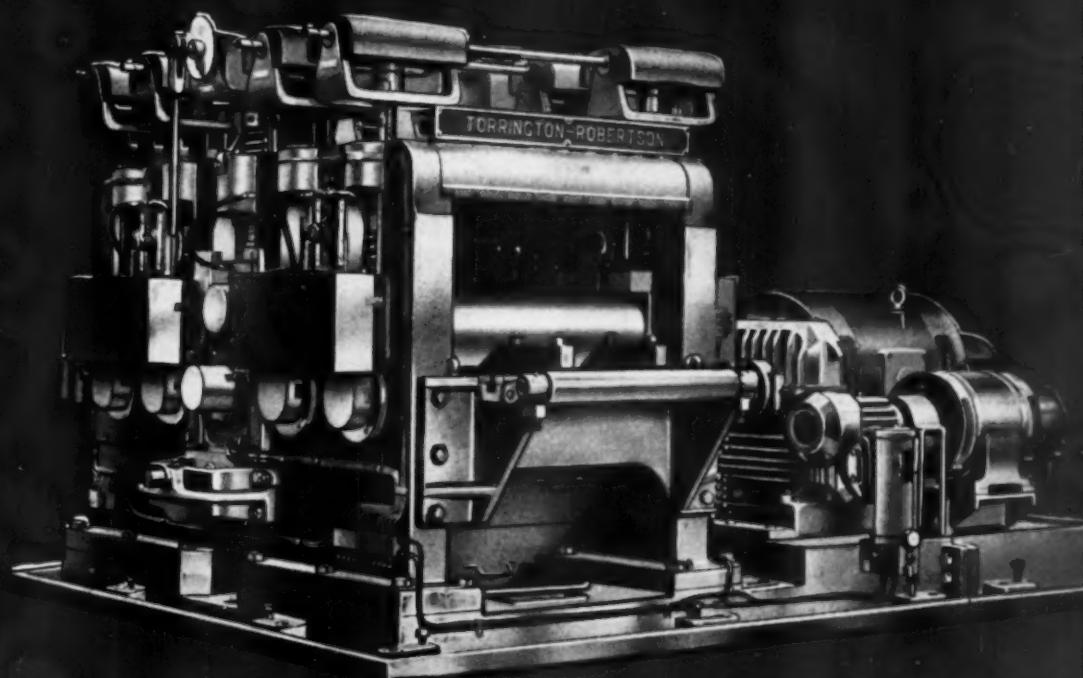
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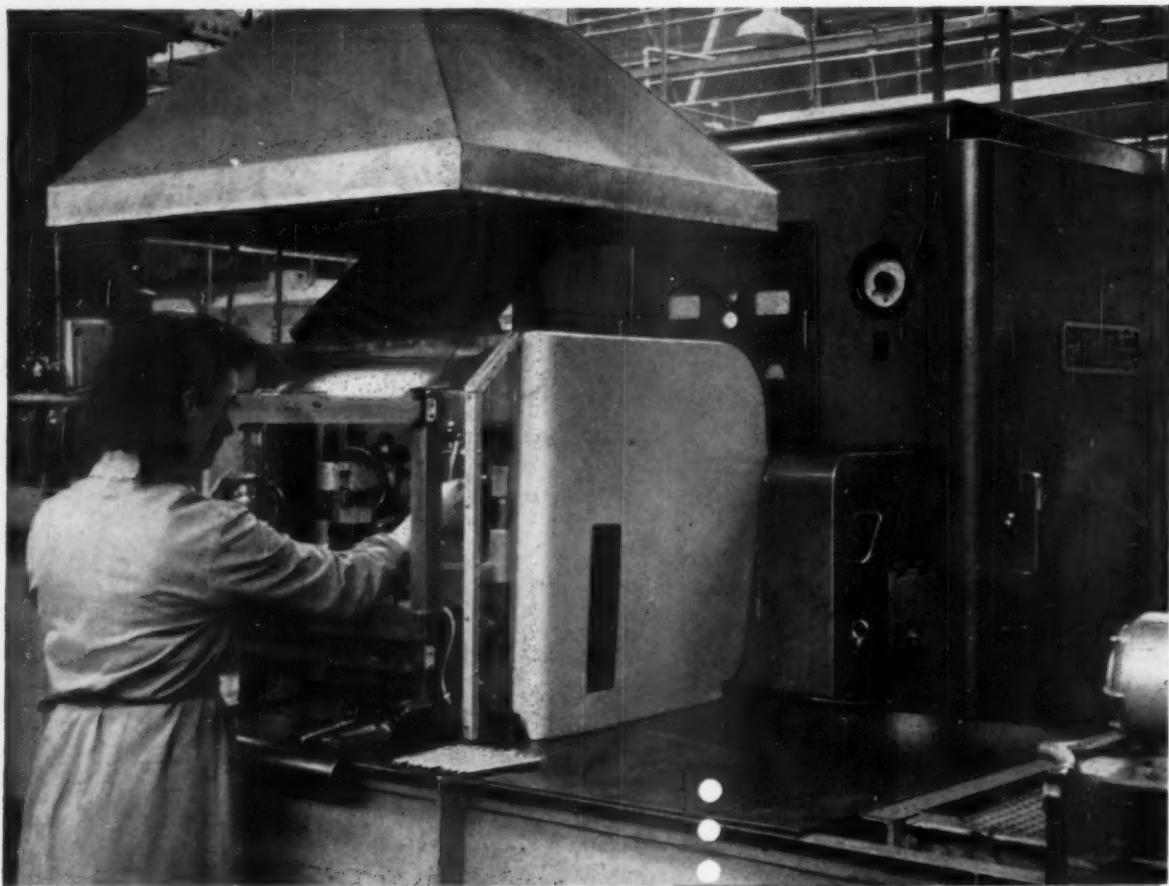


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